

STUDENT INFORMATION SHEETS

FOR SENIOR SECONDARY

CHEMISTRY PRACTICALS

FOR NATIONAL OPEN SCHOOL

H. O. GUPTA



राष्ट्रीय शैक्षिक अनुसंधान और प्रशिक्षण परिषद्
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FOREWORD

An experimental package for chemistry practicals for the open school students of senior secondary level has been developed by the NCERT in collaboration with the National Open School (NOS). It is hoped that this package will be useful not only for the Open School System but also for those senior secondary schools in the country where regular laboratory facilities may not exist due to the high cost of setting up of a chemistry laboratory. The package consists of a chemistry kit, kit manual and student information sheets. The kit contains glassware, chemicals and low cost general laboratory items. The kit manual details, inter alia, the laboratory safety regulations and teacher's notes on each tried out experiment. It is expected that the student information sheets detailing instructions for the students to do the experiments in the resource centres and to record their observations in these sheets would qualitatively improve the performance of the students in the practicals.

I thank Dr. H. O. Gupta, Reader, Workshop Department, who has made a good beginning in bringing out this experimental package for the open school students and Mrs. S. Mitra of the NOS for active association with this project as also to Prof. R. N. Mathur, Head, Workshop Department and his colleagues and all the subject experts and participating teachers for their contributions in trying out the experiments and reviewing the manuscripts.

I hope this package will be useful for enabling the students to do their practicals at low cost and in the absence of a regular laboratory. Suggestions and comments for further improvement of this package are invited.

A. K. Sharma

Director

New Delhi

National Council of Educational Research & Training

PREFACE

In the last decade, open learning systems have gained popularity in India. These systems provide access to education to drop out, women and other disadvantaged persons of the society, and are likely to expand in near future. One of the challenges faced by these systems is in implementing courses which require experimental work. In many areas of India including the remote areas, there is practically no infrastructure facilities available for experimental work. Consequently, a stop gap arrangement would be to supply compact portable and low cost kits to be placed at selected Resource Centres.

On request of the National Open School, the NCERT has taken up a project of design and development of prototype of Physics, Chemistry and Biology kits at Sr. Secondary level. The chemistry kit is the first which has been developed by the Workshop Department of the NCERT. It contains all the materials required for carrying out all the experiments prescribed by the National Open School for Senior Secondary students in chemistry alongwith kit manual and students information sheets. One kit can be used by about 18 students at one time. It is a low cost kit which could also be used in the formal system, at places where adequate laboratory facilities are not available.

The major credit for developing this chemistry kit package goes to Dr. H.O. Gupta, Reader in Chemistry in this Department. I also gratefully acknowledge the contributions of the following experts and teachers of various Institutions for the development of this package, Dr. P. S. Jaiswal, Hansraj College, Delhi; V. C. S. Rao, Sri Venkateshwara College, Delhi; Vijay Sarda, Zakir Husain College, Delhi; A. C. Handa, Hindu College, Delhi; Sanjeev Kumar, Deshbandhu College, Delhi; S. C. Sharma S.G.T.D. Khalsa College, Delhi; Bhupendra Mehta, Swami Shradha Nand College, Alipur, Brahm Prakash, DESM, NCERT, Mrs. S. Mitra, National Open School, R.C. Gaur, Mount Carmel School; Shri P. R. Juneja, HBDV Sr. Sec. School, Yusuf Sarai, R. S. Sharma, K. V. AFA Tughlakabad, Mrs. Radhika Mathur, DPS, Mrs. Pramila Jain, K.V., Majsid Moth; Sadiq Nagar and Mrs. V. K. Jetley, K.V., JNU Campus, New Delhi.

I am thankful to all the staff members of the Workshop Department, NCERT, who were involved in the development of the kit, particularly, Shri K. L. Mathur, Technical Officer, Miss Mohini Mansani and Sh. S. S. Wadhwa and Shri Anil Kumar for the art work.

R. N. Mathur
Head, Workshop Department
NCERT

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INTRODUCTION

Since chemistry is an experimental science, experiments form an integral and essential components in learning of chemistry. Before we go over to the experiments in chemistry laboratory, let us first know what does an experiment in chemistry laboratory mean. Is it just shaking a test tube or mixing solutions and seeing the colour change or something more ?

Experiment is a very broad term. Experiments are done practically in every sphere of life, at home, at place of work, in formal laboratories, etc. An experiment has five components .

- (i) Aim
- (ii) Act or performance
- (iii) Observations
- (iv) Tabulation of data
- (v) Calculation and interpretation of data

With some aim in mind, we work on apparatuses/materials to perform some activity, then make observations and record them. On the basis of known principles, we try to interpret the data and draw conclusions, for example, suppose, our aim is to estimate the amount of acid in a given solution. We perform a titration with suitable base and observe the end point and find the amount of base required for complete neutralisation and tabulate the data. Then by using the molarity equation, we make calculations and find the result.

Experiments in chemistry depend on the kind of question we ask ourselves i.e. the aim. These can be classified on the basis of the aims as follows :

- | | | |
|----|-------------------------------------------|-------------------------|
| 1 | What is it ? | Qualitative analysis |
| 2. | How much of it is there ? | Quantitative analysis |
| 3. | How do I make it in the lab ? | Preparative experiments |
| 4. | How does it behave ? | Study of properties |
| 5. | What Next ? or can I have something new ? | Synthesis experiments |

In this lab course you are going to perform experiments, the list of which along with their classification, is given in table 1.1

The experiments designed are quite safe, if done in a systematic manner as instructed. You are required to go through the instructions carefully before taking up any experiment. Convince yourself that you have understood the experiment and would be able to perform it. If required, you may consult the co-ordinator/instructor.

Before taking up any experiments, go through the general techniques/precautions to be observed in any chemistry laboratory. These are given in the subsequent sections. Specific precautions are given for each experiment. Follow them carefully.

Table 1.1 – LIST OF EXPERIMENTS

- | | | |
|-----|----------------------------------------------------------------------------------------------------------------------------------------------------|------|
| 1 | Preparation, collection and study of some important physical and chemical properties of at least three gases, one each from the following groups . | (P) |
| | (a) Chlorine, Hydrogen Chloride and Sulphur dioxide | |
| | (b) Carbon dioxide and Hydrogen Sulphide | |
| | (c) Hydrogen and Oxygen | |
| 2. | (a) Preparation of dilute solutions (5M&M/10) of Sulphuric acid, Hydrochloric acid and Nitric acid. | (P) |
| | (b) Comparison of the reactivity of metal (Cu,Fe,Zn&Mg) | (S) |
| 3. | (a) Determination of the pH of a given solution by Universal Indicator solution method. | (QT) |
| | (b) Study the change in pH of | (QT) |
| | i) acetic acid by adding sodium acetate | |
| | ii) ammonium hydroxide by adding ammonium chloride | |
| 4. | Determination of melting point of a solid substance (low melting point below 100°C) by glass capillary tube method. | (QT) |
| 5 | Study of solubility of solid substances in water at different temperatures and plotting of a solubility curve. | (S) |
| 6. | Study of neutralization reaction of (a) strong acid and strong base (b) weak acid & strong base. | (S) |
| 7. | Study the effect of (a) concentration and (b) temperature on the rate of reaction between sodium thiosulphate and hydrochloric acid. | (S) |
| 8. | Separation of coloured substances by paper chromatography and comparison of their R _f values. | (S) |
| 9. | Detection of nitrogen, sulphur or halogens in the given organic compound. | (QL) |
| 10. | Preparation of soap by using different oils. | (P) |
| 11. | (a) Preparation of standard solution of oxalic acid. | (P) |
| | (b) Standardization of a given solution of potassium permanganate by titrating against a standard solution of oxalic acid. | (QT) |
| 12. | Elementary qualitative analysis of a salt involving detection of one cation and one anion. | (QL) |

P – Preprative

S – Study of properties

QL – Qualitative

QT – Quantitative

Sy – Synthesis

If these general precautions are observed, the risk of accidents would be minimized. Still it would be worth while, if you go through the following section. It deals with common possible accidents due to improper handling of chemicals and glass ware, and their remedies i.e the first aid

1.2

SAFETY AND FIRST AID

Chemical laboratories are known for storing a number of hazardous chemicals. It is, therefore, not uncommon to face accidents in the laboratories. Every user of the laboratory must insure their and their colleague's safety. The following do's and don'ts in the laboratory would help you to avoid accidents.

The Do's

- Wear the lab coat or an apron before starting the work.
- The burner is lighted by a match stick.
- While heating a liquid in a test tube, always keep the mouth of test tube away from you body and your colleague's body
- You must ensure that laboratory is equipped with a fire extinguisher and you must know how to use it.
- Always use a goggle to protect your eyes while doing sodium ignition or such other dangerous operation.
- All reactions involving pungent or obnoxious fumes be carried out either in the open or in a fuming cupboard.
- Wash your hands thoroughly with soap before leaving the laboratory

The Don'ts

- Don't wear loose clothes or synthetic clothes while working in the laboratory.
- As a general rule, never taste any chemical in the laboratory.
- Don't pipette out dangerous liquids by sucking with mouth.
- Don't insert forcibly a thermometer or a glass tube through a cork.
- Don't inhale any gas deeply and directly. It may cause suffocation. Keep the test tube a little away from your nose.
- Don't keep inflammable substances like sodium near water
- Don't keep inflammable solvents like petrol, ether, etc. near burner
- Don't add pumice stone in a boiling liquid.
- Don't touch electric switches with wet hands.
- Don't perform an unknown experiment in the laboratory alone.

However, inspite of taking all the precautions, accidents do happen. For such an eventuality one must be fully equipped with the necessary aids to remedy the injury. Apply following remedial measures in case of an accident.

REMEDIES

i) (a) CHEMICAL REAGENTS BURNS

| CHEMICAL | NEUTRALIZING WASH |
|-----------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------|
| Acids like HCl, HNO ₃ H ₂ SO ₄ | NaHCO ₃ or 2M ammonium carbonate (it does not leave a residue on clothes). Apply any soothing cream |
| Alkalies, KOH, NaOH etc. | 1M acetic acid, then apply vaseline or soothing cream. |
| Sodium | Remove solid piece and wash thoroughly with water & dilute acetic acid. |

(b) LIST OF HAZARDOUS CHEMICAL & THEIR EFFECTS

You should know the hazardous chemicals and the effects. Try not to expose yourself excessively to these chemicals.

| HAZARDOUS CHEMICALS | EFFECTS |
|------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------|
| H ₂ S | Almost as poisonous as HCN. Exposure dulls the sense of smell. |
| H ₂ SO ₄ , HF, SO ₂ , NO ₂ , Cl ₂ , Br ₂ , I ₂ , HNO ₃ | All are dangerous. When concentrated, they corrode skin HF is in particular dangerous. |
| Salts of Ag, Ba, Hg, Ni, Pb, CrO ₄ ²⁻ and MnO ₄ ⁻ | These are dangerous only when swallowed. AgNO ₃ causes caustic burns. |
| Chlorinated alkanes e.g CHCl ₃ , CCl ₄ | Most of these are narcotic and suspected carcinogens. |

- ii) **CUTS :** In chemistry laboratory, the commonest accidents are cuts from glass wares which are broken while being used . Wash the cut with cold water and if bleeding does not stop, then apply pressure on the cut, then apply antiseptic cream and a proper dressing.
- iii) **BURNS :** These are caused by hot equipment. Wash the burns with cold water for about 10 min (till burning stops), then apply burnol
- iv) **FIRE :** A fire in a beaker is extinguished by covering the glass beaker with a watch glass or a metal plate. If clothes catch fire, then lie down on the floor and roll or cover the body by a blanket.
- v) **POISONING :** If accidentally someone swallows a poisonous chemical, then let

him drink lot of fresh water if the person is still conscious If substances swallowed is corrosive, then a solution of calcium hydroxide antidote should be given only in the case of non-corrosive poison.

- vi) **EXPLOSION :** Sometimes a faulty technique at the time of doing an experiment leads to an explosion. You should always work with highly explosive chemicals or highly oxidising chemicals under strict supervision.

1.3

ASSEMBLY OF EQUIPMENT

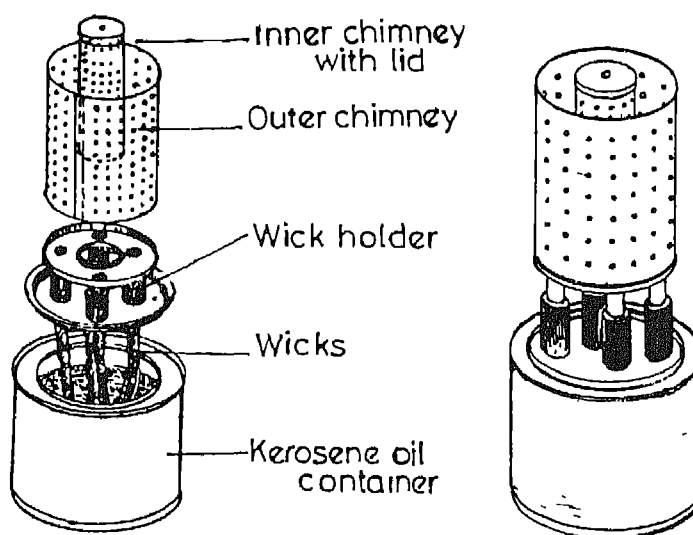
(i) ASSEMBLY OF KEROSENE BURNER

Burner has four components as shown in the diagram (Fig. 1 1)

- A. Kerosene container
- B. Wick holder
- C. Cylindrical mesh (Inner chimney) of narrow diameter
- D. Cylindrical mesh (Outer chimney) of wider diameter.

Following steps are followed for the assembly of burner

- (i) Fill the Kerosene container with Kerosene
- (ii) Fix the wicks in the wick holder
- (iii) Now fix this wick holder over the kerosene container
- (iv) Place narrow cylindrical mesh with lid to cover the wicks
- (v) Finally, fix the wide cylindrical mesh over the narrow cylindrical mesh.



1 1 Kerosene Burner and its Components

INSTRUCTIONS FOR USE

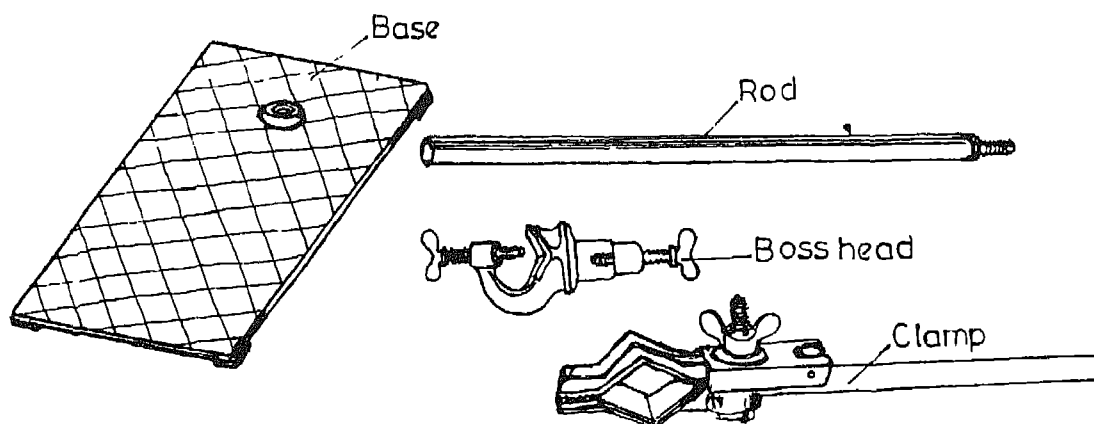
Remove both the cylindrical mesh and check that all the four wicks are equally projected out of the holder. Now, replace the narrow cylindrical mesh. Light the wicks with a match stick carefully. Put back the outer cylindrical mesh.

After finishing the work, the burner is put off by putting either a wire gauge or a flat iron plate on the top of the wire meshes and waiting till the burner extinguished and cools.

(ii) ASSEMBLY OF STAND

Components of the stand are : (Fig 1.2)

- A. Heavy metal base with hole
- B. An iron rod with thread
- C. Boss head
- D. Clamp extension



1 2 : Laboratory Stand Components

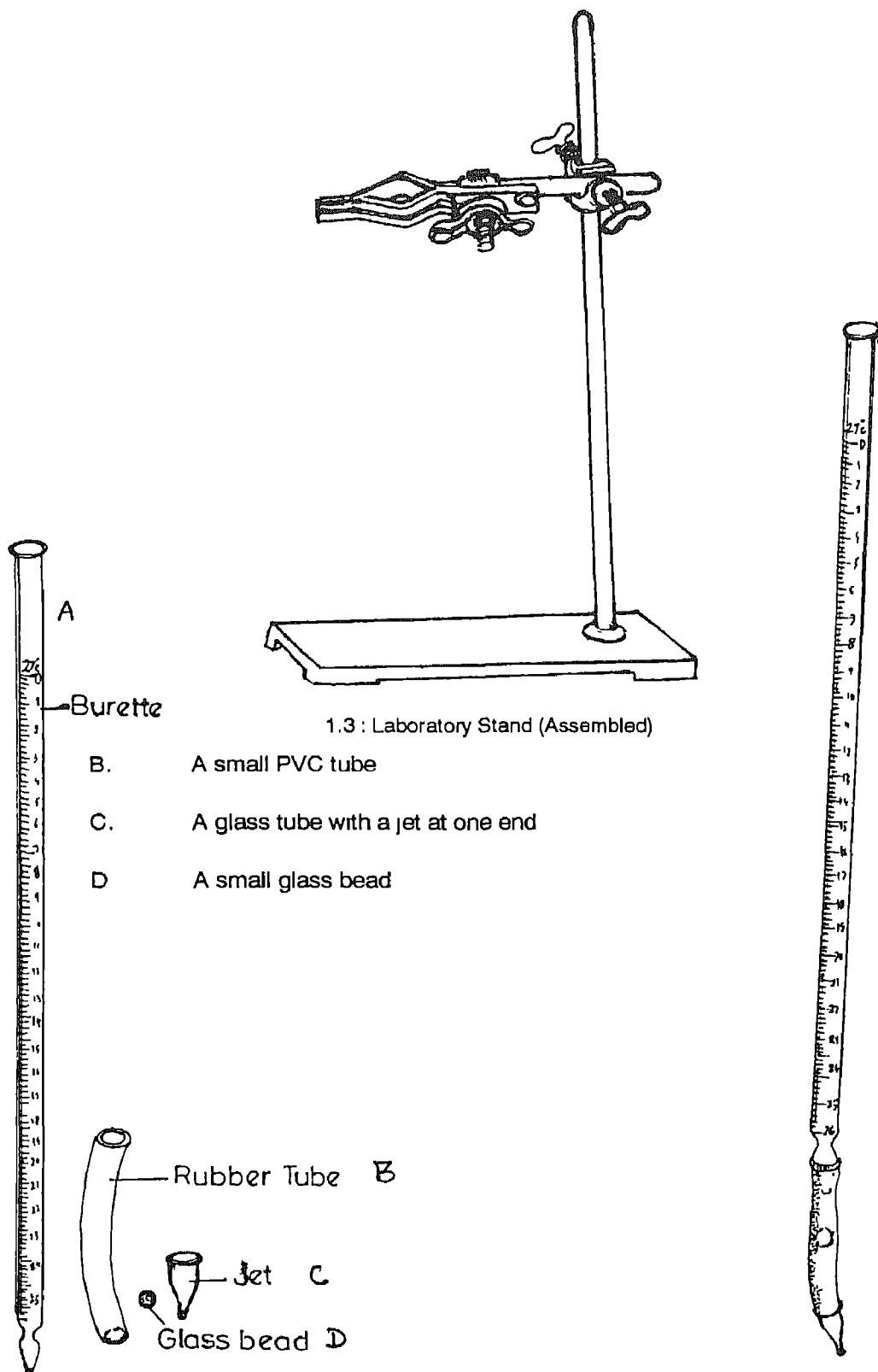
Following steps are carried out for the assembly :

- i) Place the metal base on a flat surface
- ii) Hold the iron rod with thread vertically into the hole of the base.
- iii) Now rotate the iron rod clockwise until it is tightly fixed into the base.
- iv) Now fix the boss into the iron rod at a convenient height with the help of screw.
- v) Finally fix the clamp extension into the boss as shown in the figure 1.3

(iii) ASSEMBLY OF BURETTE

Main components of the burette are : Fig. 1.4 a

- A. A hard glass tube of uniform cross section and 25.0 cm^3 capacity with division to measure 0.1 cm^3



1.3 : Laboratory Stand (Assembled)

- B. A small PVC tube
- C. A glass tube with a jet at one end
- D. A small glass bead

1.4 (a) : Burette with parts

1.4 (b) : Assembled Burette

Following steps are carried out for the assembly :

- i) Insert the glass bead into the PVC tube so that the bead is in the middle position.
- ii) One end of this tube is slipped over the wider side of the glass jet.

- iii) This assembly at (ii) is then connected to the narrow end of the long glass tube through the other end of PVC tube (fig 1 4b)

INSTRUCTION FOR USE

For releasing the liquid from the burette, just press the PVC tube at the place of the bead with your fingers.

(iv) ASSEMBLY AND USE OF APPARATUS TO PREPARE GASES

Gases lighter than air are collected by the downward displacement of air. Gases heavier than air are collected by the upward displacement of air. Finally, gases which are insoluble in water are collected over water.

To assemble the apparatus for gas preparation, following pieces of apparatus are needed :

- A. Boiling tube with side tube (commonly called filtration tube)
- B. Thistle funnel
- C. Cork bored with single hole
- D. Plastic tubing
- E. Plasticine or wax
- F. Plastic trough
- G. Beehive shelf
- H. Stand
- I. A small glass tube bent at 90°

INSTRUCTIONS FOR ASSEMBLY FOR GAS PREPARATION :

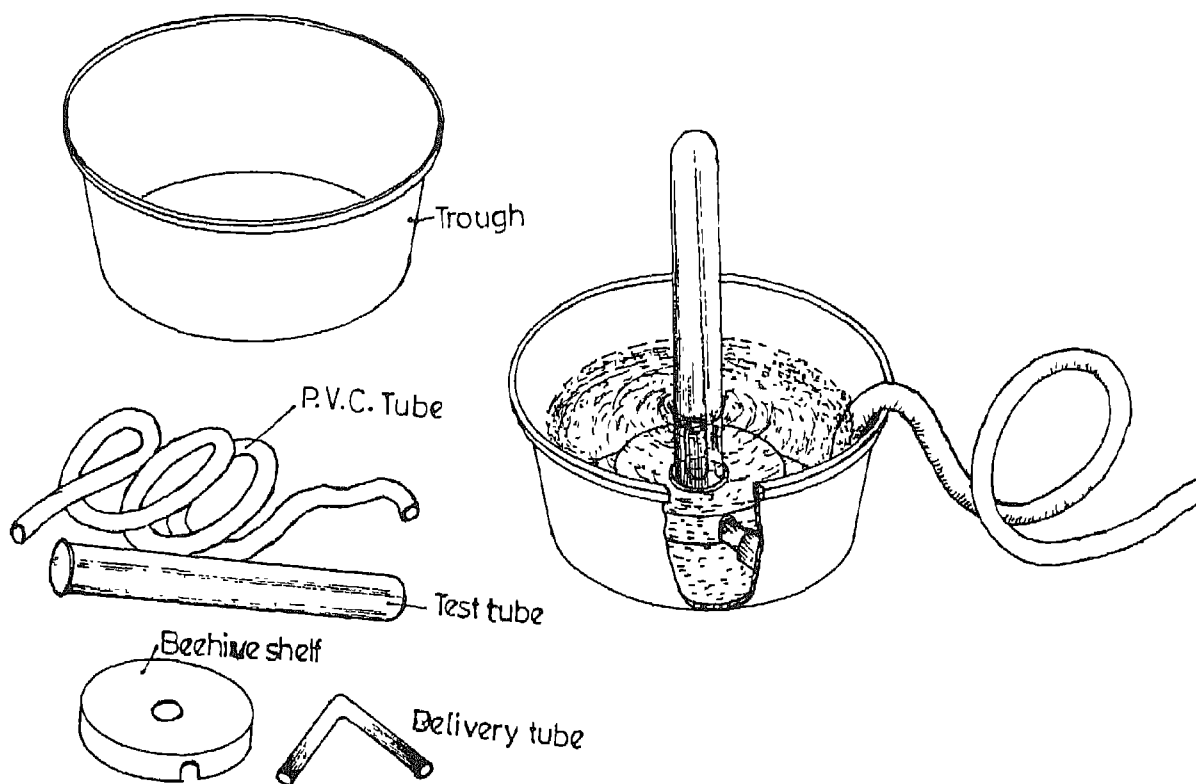
Insert the thistle funnel into the cork, fitted into the boiling tube with side tube. Now with plasticine or wax, plug the air gaps between the mouth of test tube and cork to prevent the escape of gas. Clamp the boiling tube with thistle funnel to the stand. Connect the side tube of the boiling tube and the bent glass tube through a plastic tube.

COLLECTION OF GASES

(a) GASES INSOLUBLE IN WATER

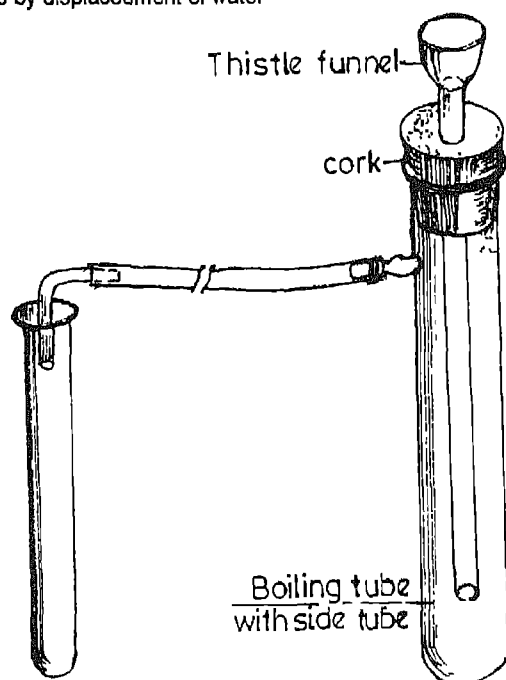
Take the plastic trough and place the beehive shelf at the centre of the trough. Fill the trough with water till it covers the beehive shelf. The apparatus and assembly for the gas collection in water is shown in figures 1.5(a) and (b) respectively. Separately fill the test tube in which gas is to be collected with water and keep it ready. Take the necessary chemicals for preparing a gas in the boiling tube. Press the mouth of the test tube full of water with your thumb and lower it in inverted position over the beehive

shelf such that water does not flow out of the test tube. Now heat the boiling tube and collect the gas by the downward displacement. After the test tube is full of gas, remove the test tube while keeping its mouth downward and cork it. Fill required number of test tubes in a similar manner.

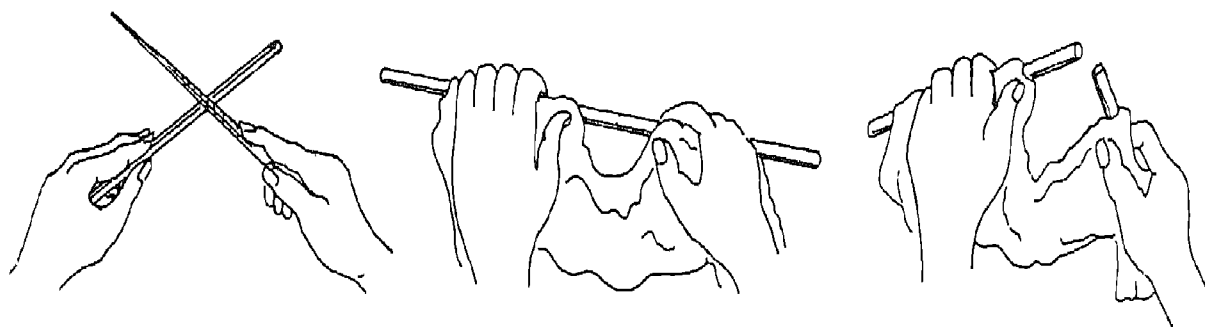


1.5 (a) : The components of assembly for collecting the gas by displacement of water

1.5 (b) : Assembly



1.5 (c) : Assembly showing the collection of gas heavier than air

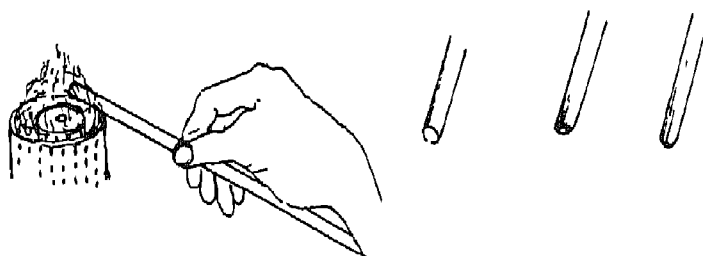


1.6 (a) . Cutting of a glass tube 1.6 (b) . Holding the glass tube 1.6 (c) Breaking the glass tube

4 Press the tube gently outward. The tube will break into two pieces Fig. (1.6c)

5. If tube does not break repeat scratch at the same point and break it by the same method.

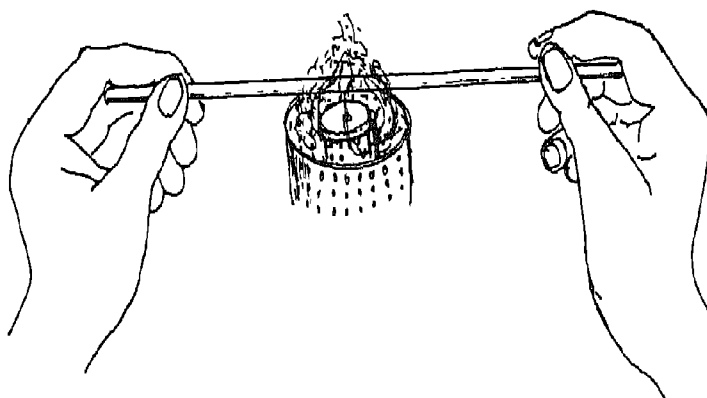
6. The freshly cut ends are sharp which may cause injury to the hands. Round off the ends by rotating the tube end in the flame for a short time. Fig. (1.6.d)



1.6 (d) Rounding off the ends of the cut tube

B. *BENDING OF A GLASS TUBE*

1. Rotate the portion of the glass tube to be bent in the flame of the burner. (Fig 1.7)

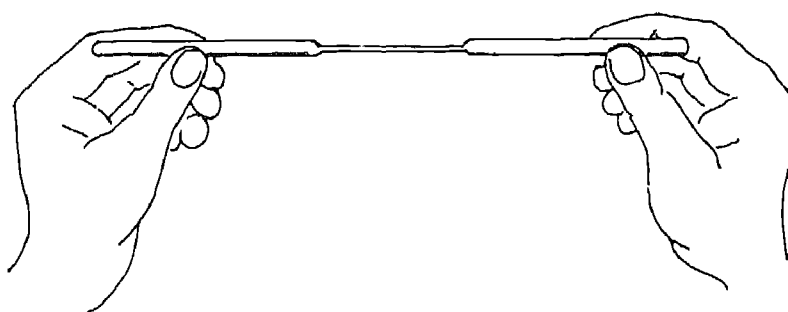


1.7 . Bending of a glass tube

2. Heat till the glass tube becomes soft enough and tends to bend by its own weight
3. Maintain the desired angle by holding the glass tube ends with both hands
4. Press the bent limbs to make them coplanar.

C *DRAWING A JET FROM GLASS TUBE*

1. Take a glass tube of a suitable length and round off its sharp edges by gently heating for some time.
2. Hold the glass tube horizontally on flame and rotate it with both hands till it becomes soft (Fig 1.8)

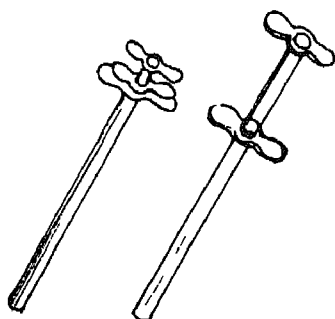


1.8 : Making a jet from the glass tube

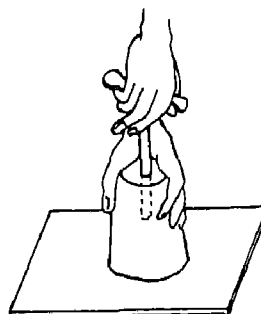
3. Withdraw the softened hot tube from the flame and draw the two ends apart (Fig 3.2c) and hold till it cools down
4. Place it on table and cut it into two jet with the help of glass- cutting file. Round off the sharp edges in the flame for short while.

D *BORING A CORK*

1. Take a good cork free from cracks and holes. Check that it should fit tight in the apparatus for which it is needed.
2. Wet the cork with water and soften it by rolling under your shoe.



1.9 (a) : Set of borers



1.9 (b) : Boring a cork

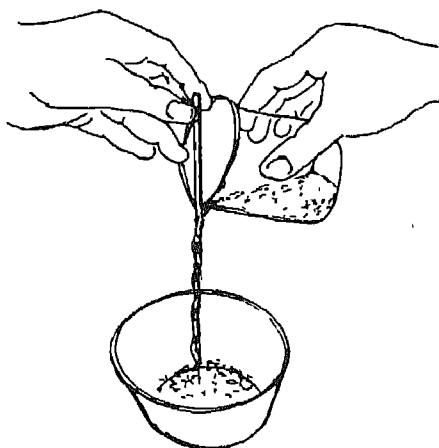
3. Now select a cork borer whose diameter is slightly less than that of the glass tube or rod for which the hole is needed
4. Place the cork on the table with its narrow end upward and hold it with in the left hand
5. Make a round hole in its centre by pressing the borer gently inward Fig 1.9(a) and (b)
6. Make a similar mark also on the opposite side of the cork. Dip the cork borer into the water, remove it and place it vertically over the mark on the cork
7. Press it downward with twisting motion into the cork till borer reaches to the half way. Pull out the borer from narrow end and remove the cork material in it by means of a needle provided with the set of borers.
8. Now place the cork upside down. Again insert the borer in cork through the mark till it meet already made hole.
9. While boring, take a precaution that hole should be straight. While inserting the glass tube into the hole, hold the cork in hand and push the tube gently through it with a rotatory motion. To avoid accidental breakage, lubricate the tube with water or soap solution.

E. *SEPARATION OF PRECIPITATES*

In the chemical reactions, an insoluble solid, called a precipitate, can easily be removed by the decantation or gravity filtration.

(a) *DECANTATION*

1. Allow the precipitates formed to settle to the bottom of container, until the liquid above is transparent
2. Hold the glass rod vertically with the left hand and touch it to the container.



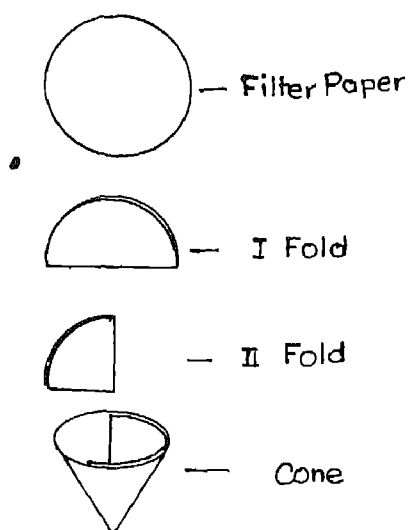
1.10 : Decantation method

3. Pour the liquid carefully to another container by tilting it (Fig 1.10) along with the glass rod
4. The holding of glass rod vertically helps in transferring the liquid to other container without any spilling and the precipitates are also not disturbed.

(b) GRAVITY FILTRATION

In the gravity filtration, the mixture containing the precipitate is poured through a filter system comprised of a conical funnel lined with filter paper.

1. Take a circular filter paper and give four folds to it. Then hold the three folds together and press a little from the corners. The filter paper acquires a cone shape as shown in figure 1.11(a).

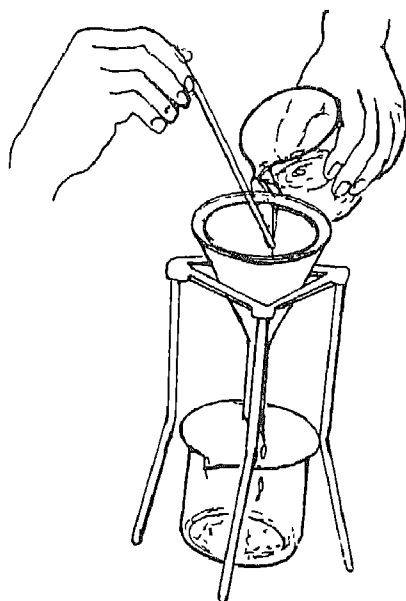


1.11 (a) : Folding of filter paper

2. Place the filter paper in the funnel and fix it on the stand or on the tripod
3. Keep another container below the funnel to collect the filtrate.
4. Transfer the liquid along with precipitate with the help of a glass rod, as mentioned earlier, to the funnel Fig 1.11(b). Collect the filtrate in the container below it and remove the filter paper from the funnel and dry the precipitate.

F. USE OF BURETTE

1. Wash the burette thoroughly with water. Put a little solution to be used in it. Now hold the burette horizontally and rotate it gently so that the whole surface gets rinsed out with the solution. Drain the solution through the jet.



1 11 (b) . Filtration

2. Clamp the burette vertically, maintaining the height in such a way that the jet of the burette enters the mouth of the titration flask. This prevents the spilling of solution (coming through the jet) outside the flask, while performing the experiment.
3. Place a funnel at the mouth of the burette. Fill it with the desired solution, a little above the etched mark at the top.
4. To displace any air bubble present, release the small amount of solution by pressing the PVC tube at the position of the glass bead.
5. If necessary, fill the burette again, to maintain the solution level upto the etched mark.
6. Read the burette by holding a white paper on back and looking straight (at eye level) to the meniscus (a curved surface of liquid).
7. For all the colourless solutions, read the lower meniscus and for the coloured solutions (e.g. KMnO_4 solutions, etc.), read the upper meniscus.
8. During the experiment, use left hand pressing the tube at the glass bead position and hold the titration flask in the right hand.

G. *USE OF PIPETTE*

1. Wash the pipette with water.
2. Dip the pipette into the solution with the tip nearly touching the bottom of the container. Suck a little solution by putting the upper end in the mouth.
3. Remove the pipette from the mouth and close its upper end with the help of the forefinger immediately. Tilt and rotate it in such a way that the inside

surface gets rinsed out completely with the solution.

4. Remove the forefinger from the upper end and drain the solution through the lower end (i.e. tip) only.
5. Suck the solution again, a little above the etched circular mark and close the pipette immediately with the forefinger.
6. Release the solution, carefully, so that the lower meniscus, in case of colorless solution (or upper meniscus in case of coloured solutions) is at the level of the circular edged mark.
7. Introduce the tip into the titration flask and discharge the solution, but removing the forefinger. The whole solution flows automatically, except a little at the tip.
8. Do not blow out the solution at the tip, but simply touch the tip to the inner wall of titration flask 2-3 times. The part of the solution passes into the flask and neglect the remaining amount still at the tip.
9. Pipettes are dangerous, if used incorrectly, for following reasons :
 - a) Sucking of the poisonous solutions, volatile liquids, concentrated acids, alkalies, etc. should be avoided as these may cause injury due to inhalation.
 - b) The horizontal placement of pipette at the laboratory bench can result in the contamination of the mouth piece. So, always keep the pipette vertically in a stand.

H. *USE OF TITRATION FLASK*

1. Wash the titration flask (a conical flask of convenient size) thoroughly with water, but never rinse it with any of the test solutions.
2. Pipette out the solution into the titration flask and keep it below the jet of the burette, preferably by holding it with the right hand.
3. Put a white glazed paper (may be torn from a magazine) or glazed porcelain tile under the titration flask, i.e. on the base of the stand. This helps in observing any slight colour change of the solution, due to indicator, at the end point in the volumetric analysis.
4. Shake the flask continuously while performing the titrations.
5. Before every titration, wash the flask thoroughly with water.

I. *HEATING OF SOLUTIONS/LIQUIDS*

Heating of a solution is done for the following purposes

- (a) concentrating the solution
 - (b) mixing or dissolving the solid components in a solvent
 - (c) for carrying out the reactions at higher temperatures.
1. Carry out the heating in any glass apparatus depending upon the requirement (i.e. in test tubes, conical flask, china dish etc.)
 2. While heating 'bumping' takes place which may cause spilling of liquids. Add a few small pieces of porcelain to avoid it. (e.g. broken china dish, cups etc.)
 3. If heating the solution is carried out in the test tube, fill it not more than 1/4. Hold it at angle of about 45° and heat on a small flame with continuous shaking.
 4. For heating of solution in beaker, china dish, conical flasks, etc., place a wire gauge on the tripod stand and keep the container over it.
 5. For heating small amount of solutions to dryness, heat them in a china dish.

STUDENT INFORMATION SHEETS

You are provided with the information sheets for each experiment. Go through the instructions and precautions carefully. Insert the data of your experiment in the blank spaces of the observations table and calculate/draw the inferences.

1a.

PREPARATION, COLLECTION AND STUDY OF SOME IMPORTANT PHYSICAL AND CHEMICAL PROPERTIES OF CHLORINE/HYDROGEN CHLORIDE/SULPHUR DIOXIDE GAS.

MAJOR SKILLS

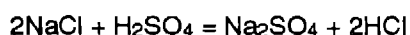
1. Setting up the apparatus
2. Observing the properties of the gas.

REQUIREMENTS

Boiling tube with side-tube, thistle funnel, soft rubber cork with one hole, 5 test tubes with corks, test-tube stand, kerosene burner, wire-gauge, tripod stand, plasticine, jet tube, plastic tube, sodium chloride salt, concentrated sulphuric acid, silver nitrate solution, litmus paper, ammonium hydroxide, match stick, laboratory stand with clamp, spatula.

INTRODUCTION

You are given the instructions for preparing hydrogen chloride gas only. Hydrogen chloride gas is prepared in the laboratory by heating sodium chloride with concentrated sulphuric acid. The chemical equation is as follows :

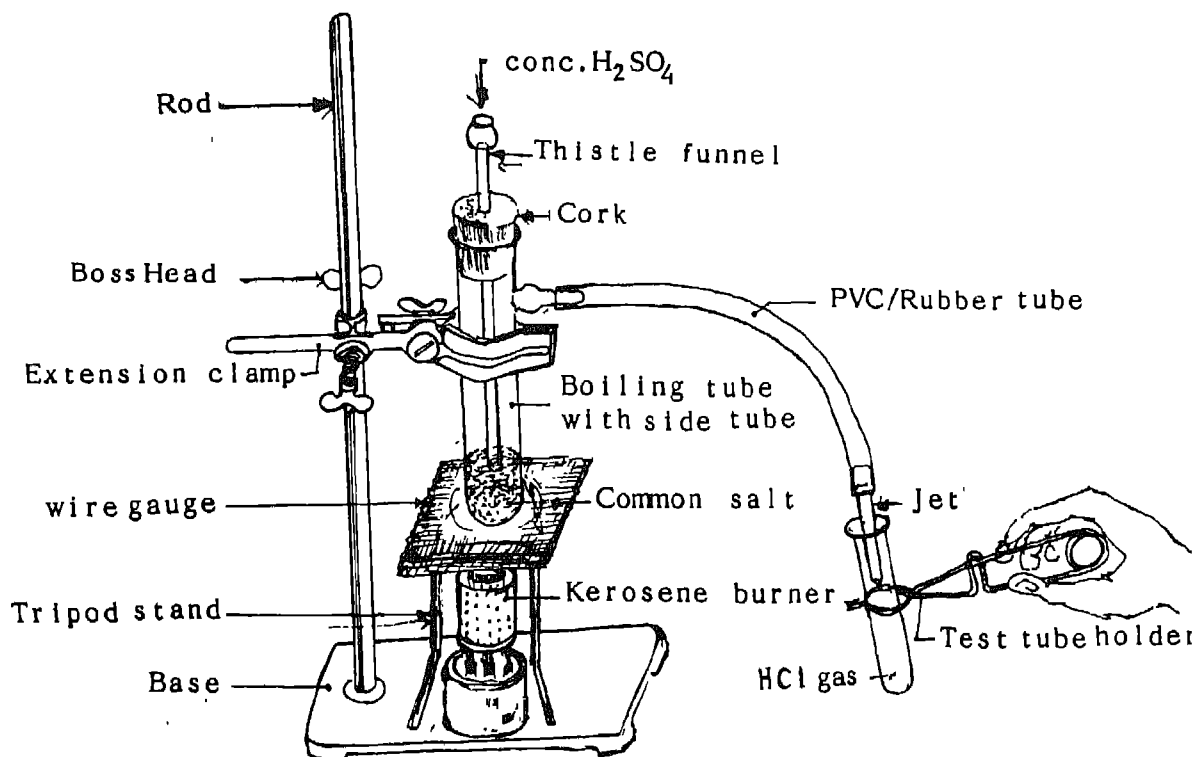


INSTRUCTIONS

1. Put about 2g sodium chloride in the boiling tube with side tube with the help of a spatula and set up the apparatus as shown in figure 1a.
2. Pour concentrated sulphuric acid through the thistle funnel such that the lower end of the thistle funnel is dipped in the acid. Make the apparatus air-tight with plasticine.
3. Take 5 test tubes in the test tube stand and label them A, B, C, D and E.
4. Heat the boiling tube gently and collect the gas in test-tubes A, B and C by upward displacement of air and cork them.
5. Take some water in test-tube D and pass the gas through it.
6. Take about 2 cm³ solution of AgNO₃ in test tube E and pass the gas in the test tube.

PRECAUTIONS

1. The apparatus should be air-tight.
2. Do not inhale the gas.
3. The test-tube should be dry.
4. Lower end of the thistle funnel should dip in concentrated sulphuric acid.



1a . Preparation of hydrogen chloride gas

OBSERVATION AND RESULT TABLE

| EXPERIMENT | OBSERVATION | INFERENCE |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------|
| PHYSICAL PROPERTIES 1. Colour (test tube A) 2. Odour (test tube A) 3. Solubility in water (test tube D) | | |
| CHEMICAL PROPERTIES 4. Acidic nature : Put a moist litmus paper in test tube A. 5. COMBUSTIBILITY Put a burning match stick in test tube B. 6. REACTIONS (a) Put a glass rod dipped in NH_4OH solution near the mouth of test tube C (b) Pass the gas through test tube E that has silver nitrate solution in it. | | |

CONCLUSION

The properties of hydrogen chloride gas are

QUESTIONS

1. Why is Hydrogen Chloride gas collected by upward displacement of air ?
2. Why does ammonium hydroxide give white fumes with HCl gas ?
3. Write the chemical reaction between hydrochloric acid and silver nitrate solution

1b.

PREPARATION, COLLECTION AND STUDY OF SOME IMPORTANT PHYSICAL AND CHEMICAL PROPERTIES OF CARBONDIOXIDE GAS.

MAJOR SKILLS

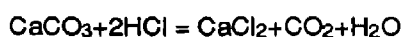
1. Setting up of apparatus
2. Learning to observe the change in colour of litmus and of certain solutions when a gas is passed through them.
3. Observing the properties of the gas.

REQUIREMENTS

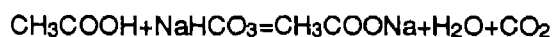
Boiling tube with side tube, thistle funnel, soft rubber cork with one hole, 4 test-tubes with corks, test tube stand, plastic tube, stands with clamp, jet tube, Marble pieces, concentrated Hydrochloric acid, red and blue litmus paper, lime water, plasticine, match stick.

INTRODUCTION

Carbondioxide gas can be prepared in the laboratory by the action of dilute Hydrochloric acid (dil HCl) on marble (Calcium carbonate - CaCO_3)



It can also be prepared by reacting acetic acid on sodium bicarbonate (NaHCO_3)

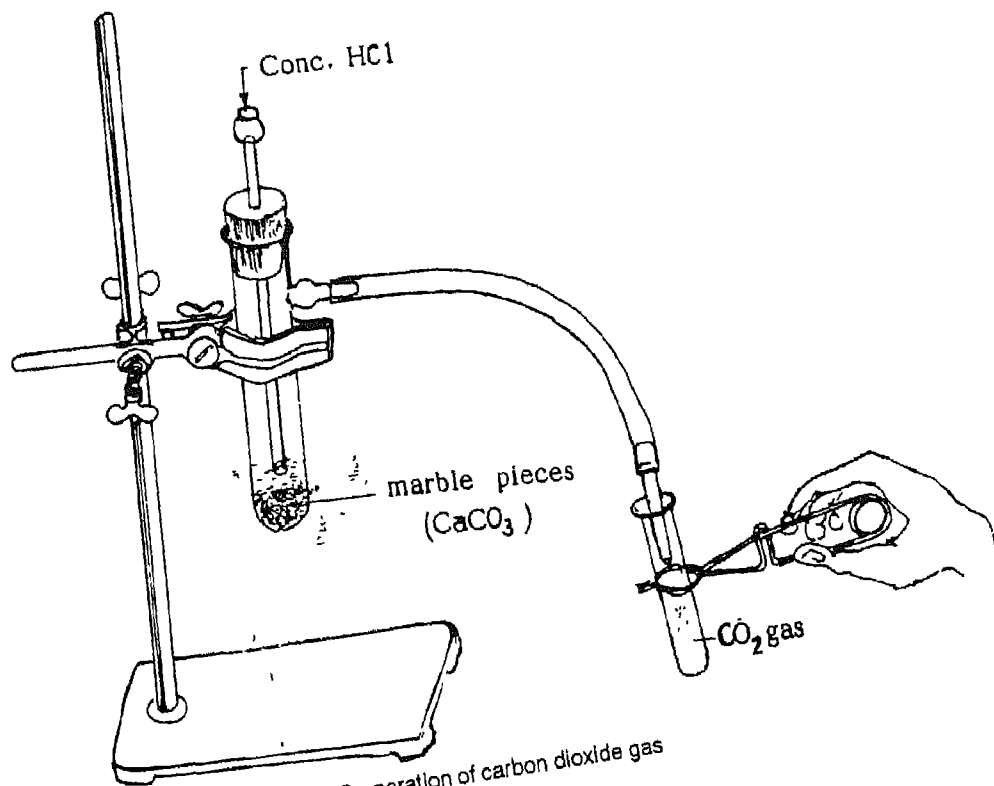


INSTRUCTIONS :

1. Put a few marble pieces in the boiling tube.
2. Set up the apparatus as shown in the figure 1b.
3. Make the apparatus air tight by sealing the cork with wax or plasticine. Take four test-tubes and label them A,B,C and D.
4. Pour some water in the thistle funnel till the lower end of the thistle funnel dips in the water.
5. Add concentrated hydrochloric acid into the thistle funnel dropwise till the reaction starts (Conc. HCl can be added whenever more gas has to be collected).
6. Pass the gas into test-tubes A,B and C and collect by upward displacement of air. Close the test-tubes with cork.
7. Take some lime water/Barium chloride solution in test tube D and pass the gas through solution. If using lime-water, pass the gas in excess and make observation.

PRECAUTIONS :

1. The apparatus should be airtight
2. Always add the acid dropwise.



OBSERVATION AND RESULT TABLE

| EXPERIMENT | OBSERVATION | INFERENCE |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------|
| PHYSICAL PROPERTIES 1. Colour (test tube A) 2. Odour (test tube A) 3. Solubility in water (test tube A) | | |
| CHEMICAL PROPERTIES 4. Introduce a burning splinter in test tube B and observe 5. Add moist blue litmus paper in test tube C 6. Pass the gas in test tube D containing lime water/barium chloride. If using limewater pass excess of gas in test tube D. | | |

CONCLUSION

The properties of carbon dioxide gas are

QUESTION :

What will happen if you try to collect the gas in water ?

1c.

PREPARATION, COLLECTION AND STUDY OF SOME PHYSICAL AND CHEMICAL PROPERTIES OF HYDROGEN/ OXYGEN GAS.

MAJOR SKILLS :

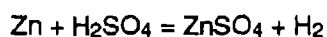
1. Setting up of the apparatus
2. Observing the properties of the gas

REQUIREMENTS

Boiling tube with side tube, thistle funnel, cork with one hole, improvised beehive shelf, plastic tube, dilute sulphuric acid, 2 test-tubes with corks, litmus paper (both blue and red), plasticine, match stick, water, test-tube stand.

INTRODUCTION

You are given the instructions for preparing hydrogen gas only. It is prepared in the laboratory by reacting dilute sulphuric acid with zinc and the gas is collected by displacement of water. The chemical equation is as follows :

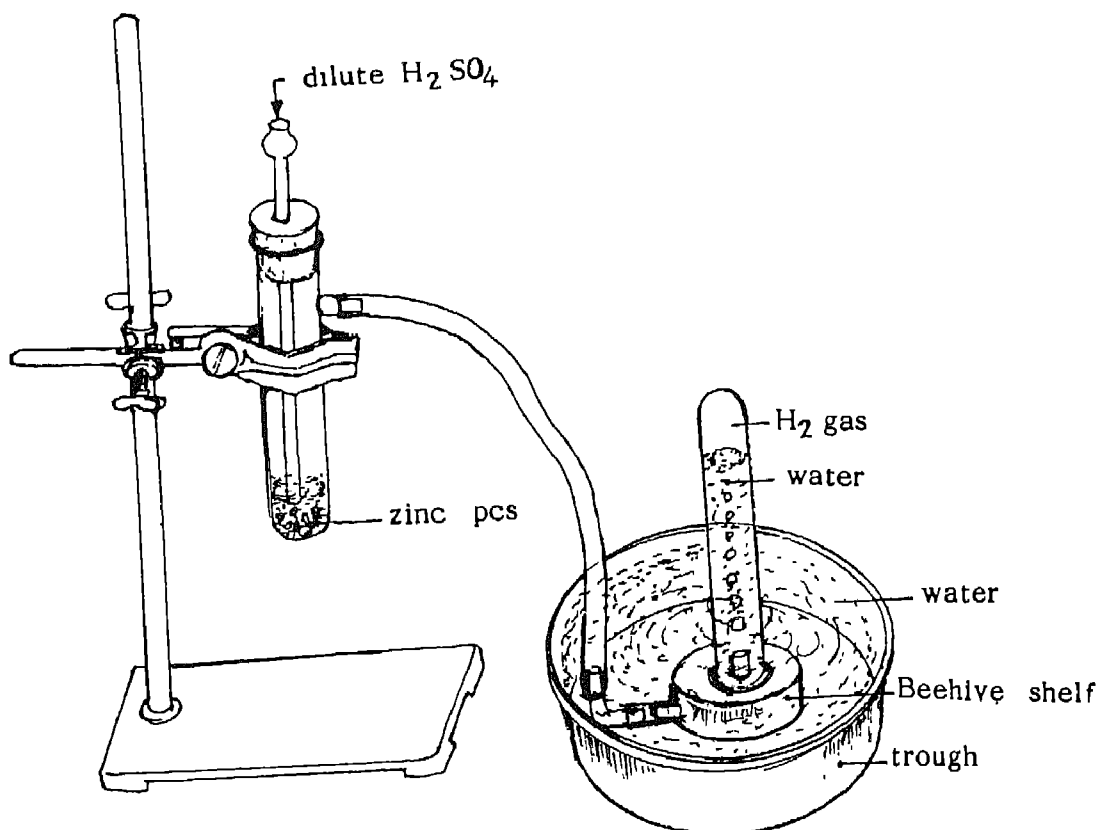


INSTRUCTIONS

1. Put a few pieces of granulated Zinc into the boiling tube with side tube. Fix the thistle funnel into the hole of the cork. Make the system air-tight by applying plasticine around the cork and thistle funnel.
2. Put a plastic tube on the side tube and lead the other end of the tube into a beehive shelf which is placed in a trough of water. A test-tube filled with water is inverted over the beehive shelf. See figure 1c.
3. Pour some diluted sulphuric acid into the thistle funnel and allow the reaction to start.
4. Collect the gas by downward displacement of water in two tubes labelled A and B.

PRECAUTIONS

- (1) End of thistle funnel should dip in dilute sulphuric acid.
- (2) Apparatus should be air-tight
- (3) Do not bring any flame near the apparatus.



1c Preparation of hydrogen gas

OBSERVATION AND RESULT TABLE

| EXPERIMENT | OBSERVATION | INFERENCE |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------|
| PHYSICAL PROPERTIES 1. Colour (test tube A) 2. Odour (test tube A) 3. Solubility in water (test tube A) | | |
| CHEMICAL PROPERTIES 4. COMBUSTIBILITY Bring a burning match-stick near the mouth of a test tube B. 5. Put moist red and blue litmus paper in test tube C. | | |

CONCLUSION

The properties of hydrogen gas are

QUESTIONS

Why is it important to keep the apparatus, for preparation of Hydrogen gas, away from flame ?

2a.

PREPARATION OF DILUTE SOLUTIONS (5M AND M/10) OF SULPHURIC ACID, HYDROCHLORIC ACID AND NITRIC ACID

MAJOR SKILLS

1. Using a measuring cylinder and volumetric flask.
2. Preparing dilute solutions of known concentration.

REQUIREMENTS

2 Volumetric flasks, measuring cylinder (100 cm^3 and 25 cm^3), wash bottle filled with water, concentrated sulphuric acid (conc. H_2SO_4), concentrated Nitric acid, (conc. HNO_3 and concentrated Hydrochloric acid (conc. HCl).

INTRODUCTION

The most commonly used acids in the laboratory are Sulphuric acid, Hydrochloric acid and Nitric acid. The concentrations of their concentrated solutions are 8M, 12M and 16M respectively. To make a required dilution, we use the equation $M_1V_1 = M_2V_2$ where

M_1 = Molarity of concentrated acid.

V_1 = Volume of concentrated acid required for dilution

M_2 = Molarity of dilute acid

V_2 = Volume of dilute acid to be prepared.

INSTRUCTIONS

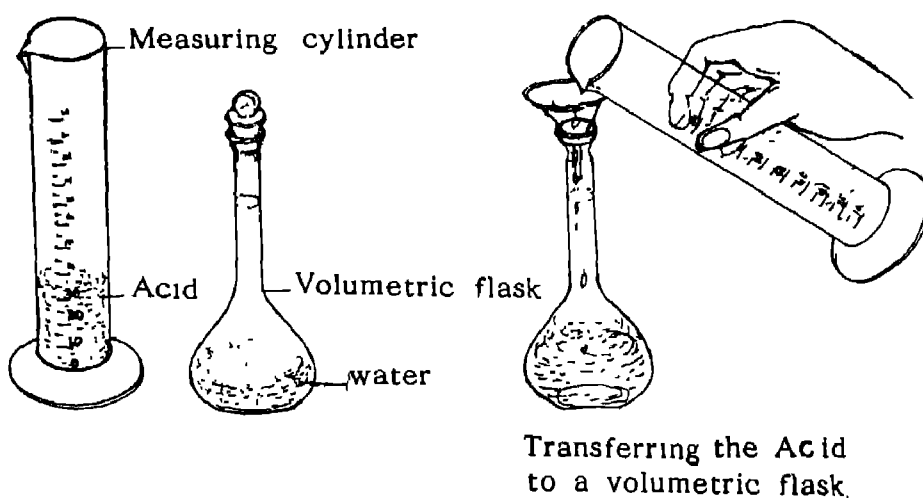
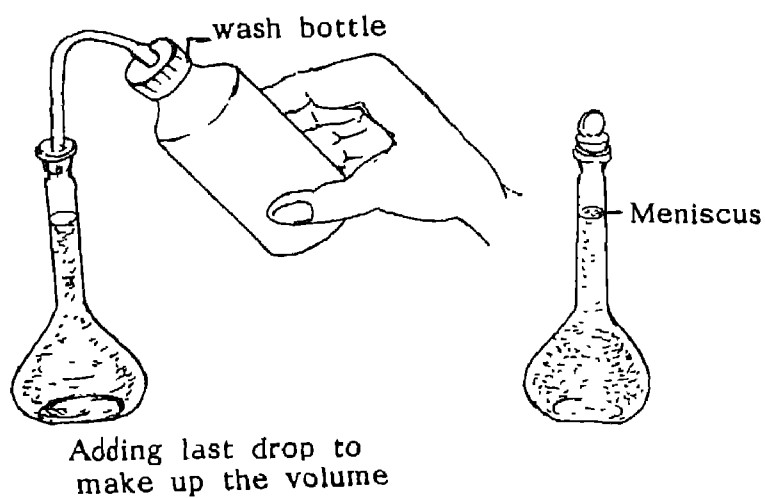
1. You are to prepare first dilute solution (5M) of one concentrated acid at one time. Take a 100 cm^3 volumetric flask and put about 50 cm^3 of water in it.
2. Take a 100 cm^3 measuring cylinder and measure the required volume of concentrated acid, 28 cm^3 of conc. H_2SO_4 , 42 cm^3 of conc. HCl , 31 cm^3 of conc. HNO_3 respectively for each dilution.
3. Pour the acid from the measuring cylinder drop by drop into the volumetric flask and stir constantly.
4. After adding all the acid add more water to make the total solution to 100 cm^3 . Label it as 5M acid (naming the acid).
5. For further dilution use 25 cm^3 measuring cylinder to take required volume of 5M acid.
6. To prepare 0.1M H_2SO_4 , 0.1M HCl or 0.1M HNO_3 , take 2 cm^3 of 5M solution of acid, respectively.
7. Repeat steps 1,3 and 4 and label the flask as 0.1M acid (naming the acid).

PRECAUTIONS

1. The acid should be added very slowly to water with constant stirring
2. Cylinder may be rinsed with little water after pouring the acid and then pouring this rinsed water into the measuring flask before making it to 100 cm^3

CONCLUSIONS

- (a) Molarity of prepared dilute solution of say, hydrochloric acid in flask/any suitable container (A) = 5M.
- (b) Molarity of prepared dilute solution of, say, hydrochloric acid in flask/any suitable container (B) = M/10.



Preparation of dilute solutions of Acid

2b.

TO COMPARE THE REACTIVITY OF METALS Cu, Fe, Zn AND Mg.

MAJOR SKILLS

1. Observing the change in colour of solution
2. Deposition of metal in a solution
3. Evolution of gas in a solution.

REQUIREMENTS

5 test-tubes, test-tube stand, pieces of Cu, Fe, Zn and Mg, Copper sulphate solution, Ferrous sulphate solution, Zinc sulphate solution and Magnesium sulphate solution, 5M dilute Hydrochloric acid.

INTRODUCTION

Metals above Hydrogen in the activity series have the capacity to displace Hydrogen from an acid e.g., Hydrochloric acid. Also a more reactive metal can displace a less reactive metal from its salt. So this experiment can be done in 2 parts.

- (a) Displacement of a less reactive metal from its salt by a more reactive metal.

If a less reactive metal is put in the salt solution of a more reactive metal, there is no reaction.

- (b) Metals above Hydrogen in the activity series can displace Hydrogen from an acid. The more reactive the metal, faster is the reaction

INSTRUCTIONS

Follow the instructions in the observation and result table.

PRECAUTIONS

Clean the surfaces of the metals with emery paper before use.

OBSERVATION AND RESULT TABLE

A. Displacement of a less reactive metal from its salt by a more reactive metal.

| EXPERIMENT | OBSERVATION | INFERENCE |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------|
| 1. Take Copper Sulphate solution (5 Cm ³ in 3 test-tubes. Add Fe, Zn, Mg pieces to different test tubes. 2. Take Ferrous sulphate solution in 3 test-tubes. Add Cu, Mg and Zn pieces to different test tubes 3. Take Zinc sulphate solution in 3 test tubes. Add Cu, Fe and Mg pieces to different test tubes. 4. Take Magnesium sulphate in 3 test tubes. Add Cu, Fe and Zn pieces to different test tubes. | | |

B. Displacement of Hydrogen from hydrochloric acid by metals. Also compare the rate of reactions.

| EXPERIMENT | OBSERVATION | INFERENCE |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------|
| 1. Take 4 test tubes and put 5 Cm ³ of 5M hydrochloric acid in each test tube. Then add pieces of Cu, Mg, Zn and Fe to different tubes. Also observe the rate of bubble formation. 2 Heat the above test tubes containing Fe, Cu and Zn And observe rate of bubble formation. | | |

CONCLUSIONS : The order or reactivity of Cu, Fe, Zn and Mg is

QUESTIONS

Why does the blue colour of copper sulphate disappear, if a piece of Mg is put in it ?

3a.

DETERMINATION OF THE pH OF THE GIVEN SOLUTIONS BY UNIVERSAL INDICATOR SOLUTION METHOD.

MAJOR KILLS

1. Measuring of pH
2. Matching of colours

REQUIREMENTS

Six test tubes, test tube stand, measuring cylinder (25 cm^3), dropper, test solutions, Universal indicator solution, colour chart.

INTRODUCTION

The concentration of hydrogen ions in a given aqueous solution determines whether the solution is neutral, acidic or basic in nature. In aqueous solution, the concentration of hydrogen ions can vary from about 1.0M to 10^{-14}M . It is quite inconvenient to represent these values. To overcome this, a term called pH has been proposed. For dilute solution, it is defined as $\text{pH} = -\log[\text{H}^+]$. The value below and above 7 correspond respectively to acidic and basic solutions. pH is accurately measured by a pH meter, but a reasonably good estimate can be made with the help of Universal indicator solution. The Universal indicator solution has characteristic colour depending on the pH of the solution.

INSTRUCTIONS

1. Take six test tubes and label them 1 to 6.
2. Put about 5 cm^3 of each of the test solutions into separate labelled test tubes. You can use 25 cm^3 measuring cylinder for this purpose.
3. To all the test tubes, add 4 to 5 drops of the indicator solution.
4. Compare the colour of the solutions in the test tubes with the standard colour chart to find the pH and record your observations in the table.
5. Classify the solutions as neutral, acidic or alkaline

PRECAUTIONS

1. Colour should be compared carefully
2. Do not add too much of the indicator solution.

OBSERVATION AND RESULTS TABLE

| S. NO. | SOLUTION | pH VALUE | CLASSIFICATION |
|--------|----------|----------|----------------|
| 1. | | | |
| 2. | | | |
| 3. | | | |
| 4. | | | |
| 5. | | | |
| 6. | | | |

CONCLUSIONS

- | | | | |
|----|---------------|-------|-------------|
| 1. | Solution Nos | ----- | are acidic |
| 2. | Solution Nos. | ----- | are basic |
| 3. | Solution Nos. | ----- | are neutral |

QUESTION

1. What will be the pH of 10^{-2} M solution of hydrochloric acid ?

You can verify your answer by adding two drops of concentrated hydrochloric acid to 100 cm^3 of water and checking its pH.

3b.

STUDY THE CHANGE IN pH OF

A) ACETIC ACID BY ADDING SODIUM ACETATE

B) AMMONIUM HYDROXIDE BY ADDING AMMONIUM CHLORIDE

MAJOR SKILLS

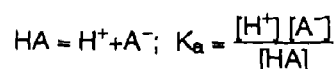
1. Measuring the pH
2. Observing change in pH of certain chemical combinations

REQUIREMENTS

5 test tubes, test tube stand, acetic acid (0.1 M), sodium acetate, colour chart, Universal indicator solution, ammonium hydroxide solution, ammonium chloride, spatula.

INTRODUCTION

Weak acid and bases do not dissociate completely. An equilibrium exists between the dissociated and undissociated forms e.g., for a weak acid, HA,



This equilibrium is characterised by a constant K_a . Addition of a salt containing common anion to a solution of weak acid causes the equilibrium to shift to the left (Le Chatelier principle). As a consequence, the concentration of hydrogen ions gets reduced and the pH gets altered. In this experiment you would study this phenomenon by using acetic acid as the weak acid and sodium acetate as the salt.

Similar arguments hold for the ionization of a weak base (e.g. NH_4OH) also. you can check this by using a salt of NH_4Cl .

INSTRUCTIONS

1. Take 5 test tubes and number them 1 to 5 and put them in the test tube stand.
2. Take 5 cm³ of a given acetic acid solution in each of the test tubes
3. In these test tubes, add varied amount of sodium acetate as given in the table. One cm³ plastic spoonful of sodium acetate weighs approximately 1 g. Dissolve it to get clear solution
4. Add 4-5 drops of the Universal indicator solution to each of the test tube.
5. Compare the colour developed in the test tubes with the colour chart and record the corresponding pH values in the table.
6. Repeat steps 1 to 5 by taking ammonium hydroxide in place of acetic acid in step 2 and ammonium chloride in place of sodium acetate in step 3.

7. The data for ammonium hydroxide/ammonium chloride system is to be recorded in table 2.

OBSERVATION AND RESULTS TABLE 1

| TEST TUBE NO | VOL. OF ACETIC ACID (CM ³) | AMOUNT OF SODIUM ACETATE (GMS) | pH |
|--------------|----------------------------------------|--------------------------------|----|
| 1 | 5.0 | 0.0 | |
| 2. | 5.0 | 0.5 | |
| 3. | 5.0 | 1.0 | |
| 4. | 5.0 | 1.5 | |
| 5. | 5.0 | 2.0 | |

OBSERVATION AND RESULTS TABLE 2

| TEST TUBE NO. | VOL. OF AMONIUM HYDROXIDE (CM ³) | AMOUNT OF AMONIUM CHLORIDE (GMS) | pH |
|---------------|----------------------------------------------|----------------------------------|----|
| 1. | 5.0 | 0.0 | |
| 2. | 5.0 | 0.5 | |
| 3. | 5.0 | 1.0 | |
| 4. | 5.0 | 1.5 | |
| 5. | 5.0 | 2.0 | |

CONCLUSIONS

The change in pH of

1. acetic acid/sodium acetate is found to be
2. ammonium hydroxide/ammonium chloride is found to be

QUESTIONS

The pH of the solutions obtained above does not change appreciably on adding small amount of acids or bases.

You can check it by adding a few drops of dilute hydrochloric acid or sodium hydroxide solution to the test tubes.

Can you suggest a reason for the same ? If not, discuss with your coordinator.

4.

DETERMINATION OF MELTING POINT OF A SOLID SUBSTANCE (LOW MELTING POINT BELOW 100°C) BY GLASS CAPILLARY TUBE METHOD.

MAJOR SKILLS

1. Setting up of apparatus
2. Reading the thermometer correctly
3. Determining melting point of a solid

REQUIREMENTS

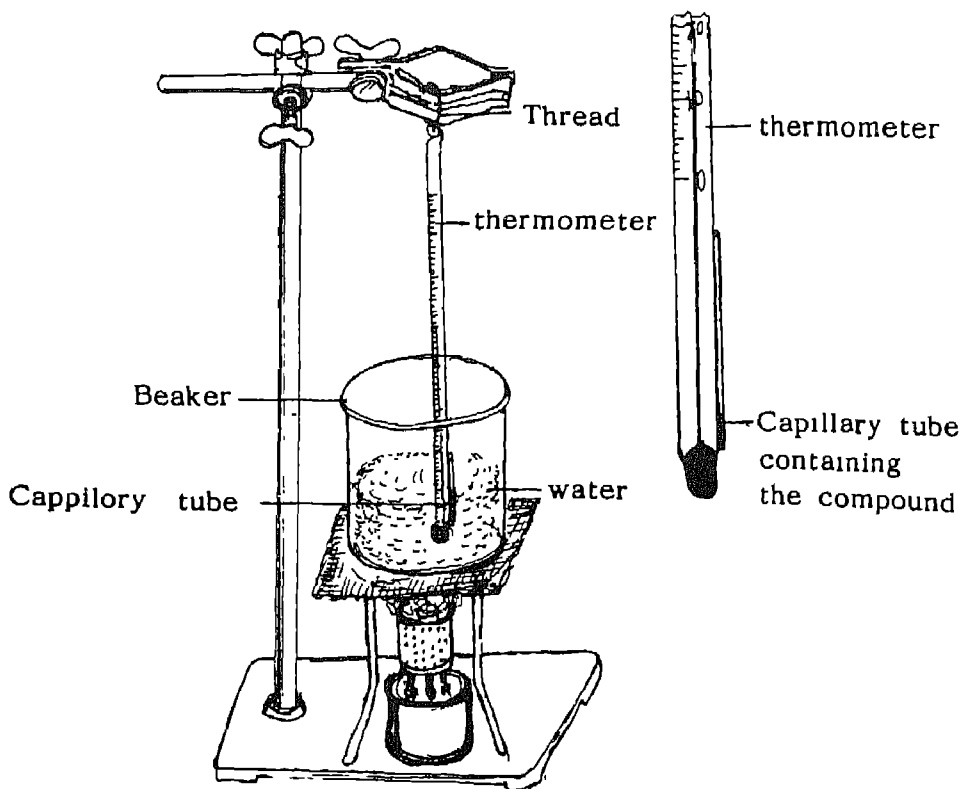
Glass beaker (100 cm³), thermometer, capillary tube (8-10 cm long), laboratory stand (with boss and clamp), tripod, burner, wire gauge, rubber band, spatula, tile, naphthalene.

INTRODUCTION

You are given a substance whose melting point is below 100°C, because water medium would be used for its melting point determination. For compounds having melting points higher than 100°C, oil or any other suitable medium having higher boiling point than the melting point of the to be compound determined is taken

INSTRUCTIONS

1. Fill about half of the beaker with water. Light the burner.
2. Take a capillary tube and close one end by holding the end in the flame for 2-3 minutes.
3. Check whether the end is sealed properly.
4. Take the given organic compound, say naphthalene, on a tile and grind it into a very fine powder.
5. Hold the closed end of the capillary between your thumb and finger. Now dip the open end in the fine powder of the organic compound. Gently tap your thumb holding the capillary tube on the table to fill the capillary to a length of about 1 cm.
6. Gently attach the capillary tube to the thermometer with the help of a rubber band. See that the bulb of the thermometer touches the closed end of the capillary.
7. Place the beaker on a wire gauge placed on a tripod.
8. Hang the assembly of thermometer and capillary from the clamp such that it dips in water fully but does not touch the bottom (See figure 2).
9. Start heating water in the beaker with the burner. Watch the closed end of the capillary carefully. Note down the temperature as soon as the substance begins



2 : Determination of melting point

to melt (first appearance of the liquid). Repeat the procedure with other capillaries two-three times and record your observations.

PRECAUTIONS

1. While filling the capillary tube, take care that the closed end does not touch the surface.
2. Bulb of the thermometer should not touch the bottom of the beaker and is about 1 cm above the bottom of the beaker.

OBSERVATION AND RESULT TABLE

| S. No. | Name of the substance | Temperature at which substance begins to melt ($^{\circ}\text{C}$) |
|--------|-----------------------|----------------------------------------------------------------------|
| 1. | | |
| 2. | | |
| 3. | | |

CONCLUSION

The melting point of IS $^{\circ}\text{C}$

QUESTION

Why do you determine the m.pt. of a substance melting below 100°C in water ?

5.

STUDY OF SOLUBILITY OF SOLID SUBSTANCES IN WATER AT DIFFERENT TEMPERATURES AND PLOTTING OF A SOLUBILITY CURVE.

MAJOR SKILLS

1. Setting up of apparatus
2. Recording the correct temperature at the time of dissolution and reappearance of solid.
3. Plotting the curve

REQUIREMENTS

Boiling tube, glass rod, copper wire stirrer, glass beaker (250 cm³) thermometer, wire gauge, tripod, kerosene burner, stand with clamp and boss, potassium nitrate, cork with two holes, 1 cm³ spatula

INTRODUCTION

Variation of solubility with temperature is quite different for different substances. For some, such as KNO₃, the solubility changes very rapidly, while for others such as KI the change is much less. These differences in solubility behaviour provide the basis for a useful laboratory technique, called fractional crystallization, which is frequently used for the separation of impurities from the products of a chemical reaction

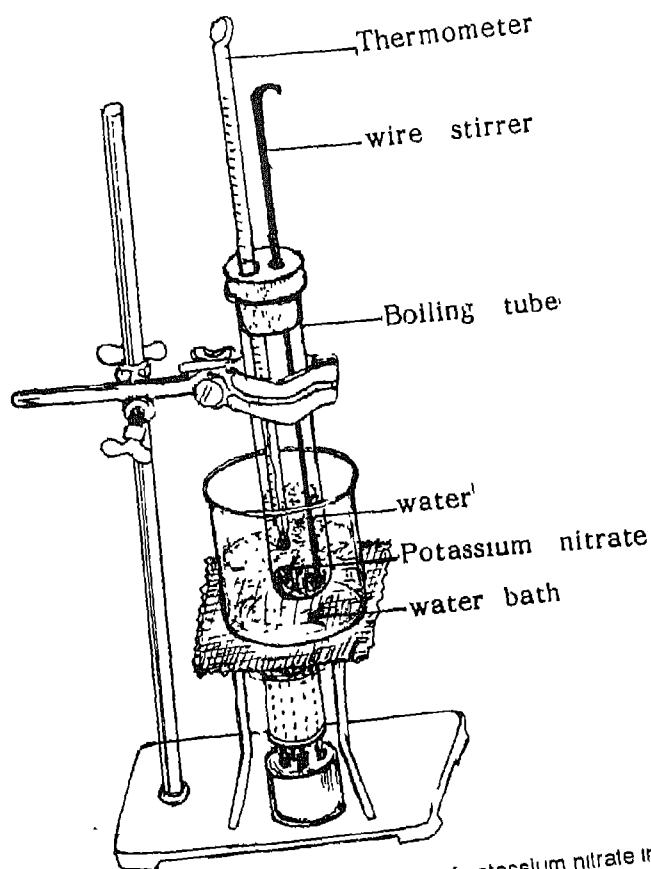
INSTRUCTIONS

1. Take 5 cm³ of water in the boiling tube. Add 7.5g of potassium nitrate to it with the help of 1 cm³ spatula. One 1 cm³ spatulaful will give you about 1.2g KNO₃
2. Set the apparatus as shown in figure 3 .
3. Heat the boiling tube in 250 cm³ glass beaker containing water to dissolve all the potassium nitrate while stirring with copper wire stirrer. Note down the temperature at which crystals disappear.
4. Stop heating the water beaker and note down the temperature at which the crystals of the potassium nitrate reappear.
5. Take the mean of these two temperatures.
6. Add 1 cm³ of water into the boiling tube. Cork it again and heat it to dissolve the whole of potassium nitrate. Note the temperature.
7. Stop heating and note down the temperature at which the crystals of potassium nitrate reappear.
8. Take the mean of the two temperatures.
9. Repeat this procedure by adding 1 cm³ of water each time successively to the same tube and recording the temperature in the manner of steps 6-8.

10. Calculate the amount of solute (KNO_3) per 100 cm^3 of solvent (water) and enter in this table.

PRECAUTIONS

During heating and cooling, stir the solution with the help of the stirrer constantly.



3. Determination of solubility of potassium nitrate in water.

OBSERVATION AND RESULTS TABLE

| S. N | QTY. OF KNO_3 (GMS) | VOLUME OF WATER (CM^3) | AMOUNT OF SOLUTE PER 100 CM^3 | TEMP. AT WHICH SALT DISSOLVES t_1 ($^{\circ}\text{C}$) | TEMP. WHEN SALT REAPPEARS t_2 ($^{\circ}\text{C}$) | MEAN TEMP. $\frac{t_1 + t_2}{2}$ |
|------|------------------------------|-----------------------------------|----------------------------------------|------------------------------------------------------------|--------------------------------------------------------|----------------------------------|
| 1. | | | | | | |
| 2. | | | | | | |
| 3. | | | | | | |

PLOTTING THE GRAPH

Draw a graph between the weight of KNO_3 dissolved per 100 cm^3 of water and temperature, taking temp. along x-axis and solubility against y-axis.

QUESTION

Why is it necessary to cork the tube ?

6.

STUDY OF NEUTRALIZATION REACTIONS OF

(A) STRONG ACID AND STRONG BASE

(B) WEAK ACID AND STRONG BASE

MAJOR SKILLS

1. Setting up of improvised calorimeter
2. Calculations of water equivalent of beaker (calorimeter) and heat of neutralization
3. Drawing conclusions regarding heat of neutralization

REQUIREMENTS

Glass beaker (100 cm^3), Glass beaker (250 cm^3) with lid, Rubber cork with two holes, Newspaper sheets, Thermometer, Glass stirrer, 1 M NaOH, 1 M HCl, 0.5 M H_2SO_4 1 M Ethanoic acid (acetic acid).

INTRODUCTION

Enthalpy of neutralization is change of enthalpy when 1 mole of hydrogen ions (H^+) are completely neutralized by 1 mole of hydroxyl ions (OH^-) in a dilute solution. For all strong monoprotic acids (e.g. HCl, HNO_3 etc) enthalpy of neutralization is same. However, for diprotic acids (e.g. H_2SO_4) enthalpy of neutralization of 1 mole of acid against a strong base is double that of 1 mole of monoprotic acid. The hydrogen ions (H^+) produced by 1 mole of diprotic acid are 2 moles of H^+ per mole. Find out if all strong acids and bases give the same value.

In case of weak acids and strong bases or vice versa, change of Enthalpy is not the same.

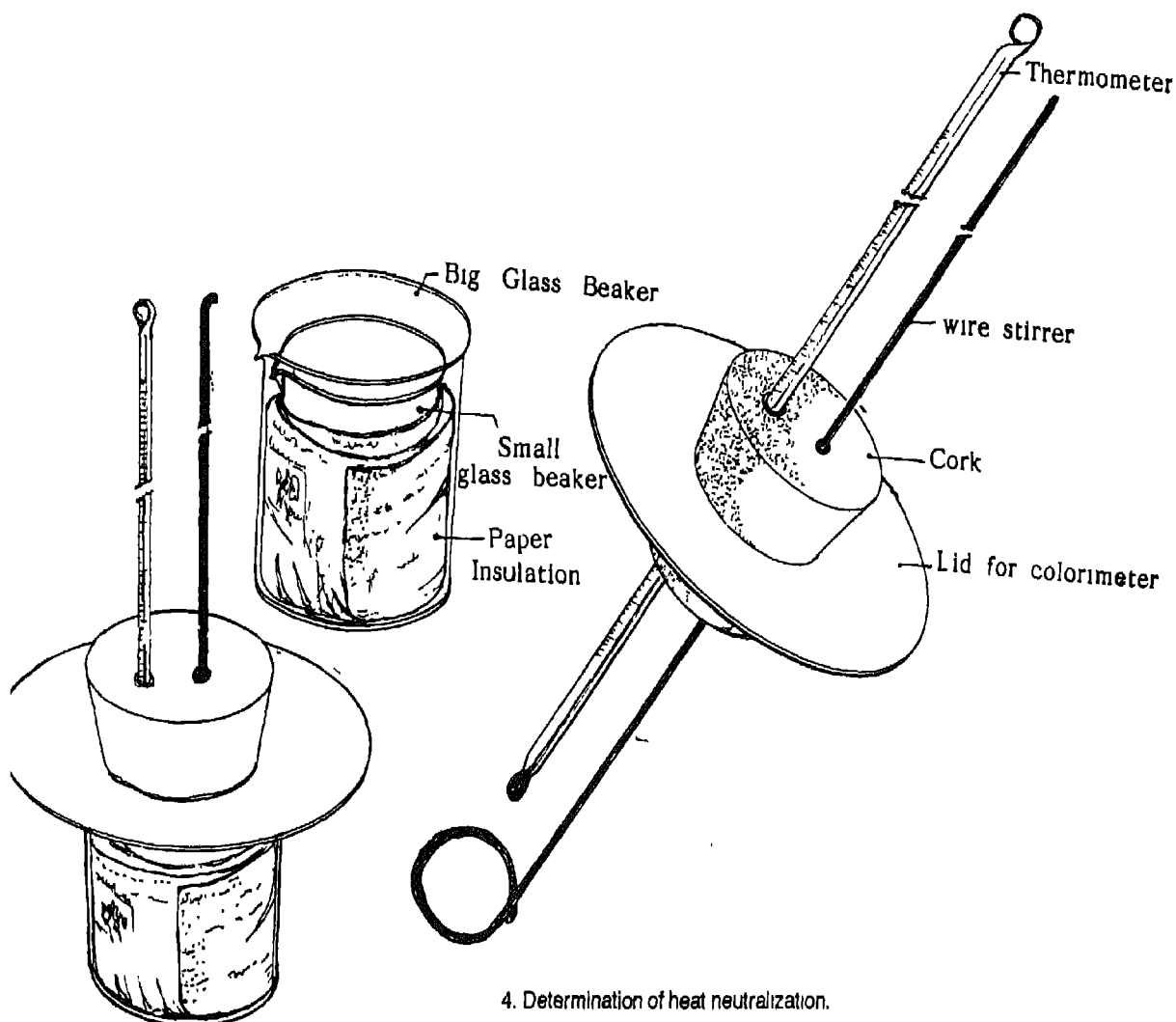
INSTRUCTIONS

1. Wrap newspaper sheets around 100 cm^3 beaker so as to fit it tightly into 250 cm^3 glass beaker.
2. Insert the thermometer and glass stirrer in the two holed cork, which is then fixed in the lid.
3. Place this lid over the mouth of 250 cm^3 beaker.
4. This assembly is your improvised calorimeter as shown in figure 4.
5. Take 20 cm^3 of water in the small beaker (100 cm^3) and note down the temperature with the help of a thermometer
6. Heat gently 20 cm^3 of water in a separate beaker to a temperature about 20°C higher than the cold water. Note down the correct temperature.
7. Remove the lid and pour this hot water quickly into cold water in the small beaker. Place the lid back. Mix by stirring for a few minutes

8. Note down the final temperature.
9. Record your observations in the table.
10. Finally empty the calorimeter.
11. Now take 20 cm^3 of 1M HCl in the calorimeter. Record the temperature when it becomes constant
12. Add quickly 20 cm^3 of 1 M NaOH into the calorimeter. Stir the mixture for a few minutes. Record the temperature.
13. Repeat steps 11 and 12 with other acid base combinations (optional).

PRECAUTIONS

1. Liquid should be stirred gently to avoid spurting.
2. Lid should always be kept on the beaker
3. After pouring the liquid into the beaker, lid should be replaced as quickly as possible.
4. Beaker should be insulated to near perfection.
5. Record the temperature correctly



4. Determination of heat neutralization.

OBSERVATION RESULT AND TABLE

| LIQUIDS | VOLUME OF LIQUID CM ³ | TEMP. (°C) | HEAT GAINED OF HEAT LOST (CAL) |
|------------|----------------------------------------|------------------|----------------------------------------|
| COLD WATER | 20 | t ₁ = | 20 (t ₃ -t ₁) = |
| HOT WATER | 20 | t ₂ = | 20 (t ₂ -t ₃) = |
| MIXTURE | 40 | t ₃ = | |
| ACID | 20 | t ₄ = | 20 (t ₆ -t ₄) = |
| ALKALI | 20 | t ₅ = | 20 (t ₆ -t ₅) = |
| MIXTURE | 40 | t ₆ = | |

CALCULATIONS

- (a) First calculate water equivalent of beaker (W) (heat gained by beaker) as follows :

Heat lost by hot water = Heat gained by beaker + Heat gained by cold water

$$20 (t_2 - t_1) = W (t_3 - t_1) + 20 (t_3 - t_1)$$

- (b) Now to calculate enthalpy of neutralization proceed as follows :

Heat evolved (Q) after mixing the two solutions,

$$Q = W (t_6 - t_4) + 20 (t_6 - t_4) + 20 (t_6 - t_5) \quad \text{Cal}$$

- (c) To calculate the heat evolved per dm³ (1000 cm³) multiply the results of (b) by 50.
- (d) Value of Q from (c) will be in cal mol⁻¹. This is converted to Jmol⁻¹ by multiplying it by 4.18.
- (e) Heat of neutralization would be expressed in negative sign.

RESULT

The enthalpy of neutralization of strong acid and strong base is ----- Jmol⁻¹.

QUESTION

The enthalpy of neutralization of acetic acid and sodium hydroxide is found to be about -45 k Jmol⁻¹. Can you suggest a reason for this comparatively low value ?

7.

STUDY THE EFFECT OF (A) CONCENTRATION AND (B) TEMPERATURE ON THE RATE OF REACTION BETWEEN SODIUM THIOSULPHATE AND HYDROCHLORIC ACID.

MAJOR SKILLS

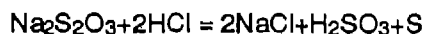
1. Observing the state of turbidity correctly
2. Measuring the volume
3. Reading the temperature value from the thermometer

REQUIREMENTS

Test tube stand, test tubes 5, measuring cylinder, stirrer, watch (student can use their own watch or the instructor may provide his watch), HCl (0.1M), Na₂S₂O₃ (0.3M), burner, match box.

INTRODUCTION

Sodium thiosulphate solution when acidified with HCl solution produces white precipitate of sulphur which causes turbidity in solution. The equation is given as under:



Time taken for turbidity to appear is taken as a measure of rate of reaction.

INSTRUCTIONS

(a) Effect of concentration

1. Take these test tubes in the test tube stand and label them 1,2,3,
2. Take 5 cm³ of sodium thiosulphate (0.3M) in the test tube 1 and add 5.0 cm³ of water.
3. Take 5.0 cm³ of sodium thiosulphate (0.3M) in the test tube 2
4. Take 10.0 cm³ of sodium thiosulphate (0.3 M) in test tube 3.
5. Now add 5.0 cm³ of HCl (0.1M) in the 1st, 10.0 cm³ in the 2nd and 5.0 cm³ in the 3rd test tube one by one.
6. In each case, note down correctly time taken in the observation table for the turbidity to appear and also time after which solution becomes opaque.

(b) Effect of Temperature

1. Take six test tubes and number them 1-6
2. Take 5.0 cm³ of sodium thiosulphate and 5.0 cm³ of water in each of the three test tubes.

3. Take 5.0 cm³ of hydrochloric acid in the other 3 test tubes.
4. These 6 test tubes are paired like 1,4; 2,5 & 3,6
5. Mix the solution of the first pair of test tubes at rooms temperature.
6. Record the time of appearance of turbidity and time in which solution becomes opaque.
7. Second pair of test tubes are heated to about 40°C and then the solutions are mixed. Again repeat step (6)
8. Repeat step (7) with the third pair of test tubes at a temperature of about 50°C.
9. Record the time in the observation table

PRECAUTIONS

1. Use identical test tubes.
2. Start the watch as soon as solutions are mixed
3. Note down the time immediately on appearance of the turbidity.
4. Time and thermometer readings should be properly coordinated.

OBSERVATION AND RESULT TABLE

(a) Effect of concentration

| S. N | Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ (Cm^3) | Vol. of HCl (Cm^3) | Time for appearance of turbidity (sec.) | Time for opaqueness of solution (sec.) |
|------|-------------------------------------------------------------|-------------------------------|-----------------------------------------|----------------------------------------|
| 1. | | | | |
| 2 | | | | |
| 3. | | | | |

(b) Effect of temperature

| S. N | Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ (Cm^3) | Vol. of HCl (Cm^3) | Time for appearance of turbidity (sec.) | Time for opaqueness of solution (sec.) |
|------|-------------------------------------------------------------|-------------------------------|-----------------------------------------|----------------------------------------|
| 1. | | | | |
| 2. | | | | |
| 3. | | | | |

CONCLUSIONS

1. Rate of reaction increases/decreases/remain constant as the concentration increases.
2. Rate of reaction increases/decreases/remain constant as the temperature increases.

QUESTION

From the results of your observations, suggest how concentration and temperature affect the rate of reaction between sodium thiosulphate and hydrochloric acid. Can you generalize it ?

SEPARATION OF COLOURED SUBSTANCES BY PAPER CHROMATOGRAPHY AND COMPARISON OF THEIR R_f VALUES.

MAJOR SKILLS

1. Setting up of apparatus.
2. Calculating the R_f values of coloured substances.

REQUIREMENTS

Measuring cylinder (100 cm³), glass rod, pin, strip of Whatman filter paper (15cmx2cm), Red and Blue ink mixture or brown sketch pen, distilled water

INTRODUCTION

This technique is used for purification and separation of compounds in a mixture. Chromatography is based on the general principle of distributing the component of a mixture between two phases, a stationary phase and a moving phase.

Here in the paper chromatography, stationary phase is water bound to cellulose (a strip of filter paper) and moving phase is normally a solvent system (mixture of solvents). In our experiment, water is only the stationary phase. As the mobile phase moves upward into the filter paper, a partition of the components takes place between stationary and mobile phase.

INSTRUCTIONS

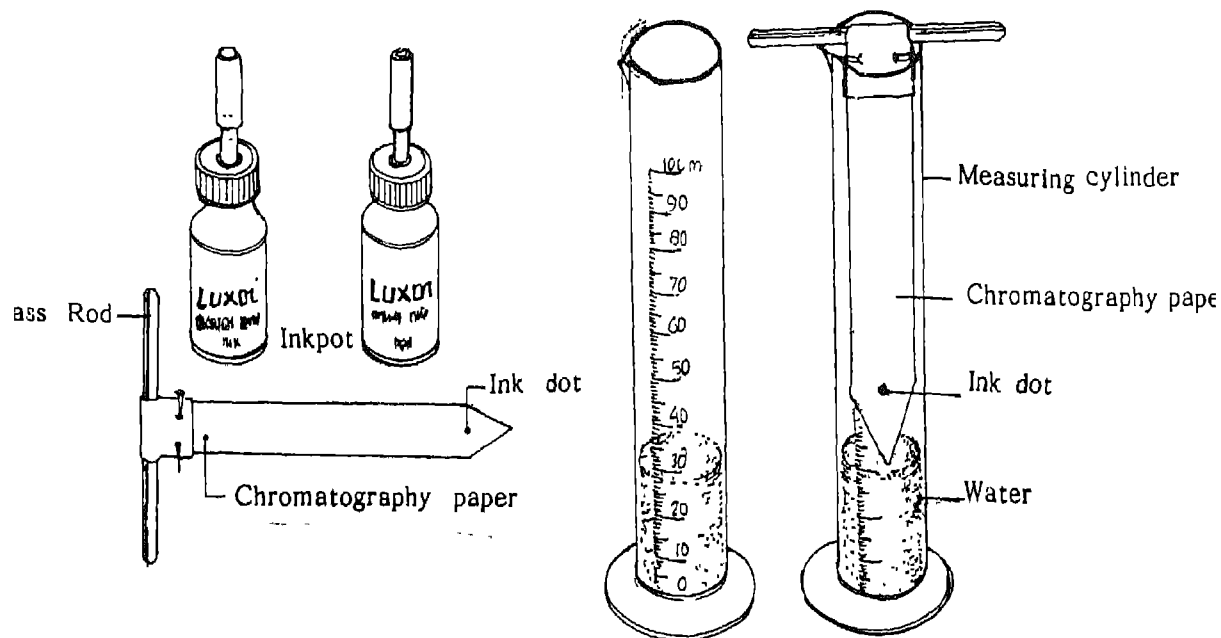
1. Take a strip of filter paper (cut from a Whatman filter paper No. 1 of the size 15cmx2cm) having one end as V shape and place it on the table. Draw a line at V shape end with a pencil (Don't use pen) at a distance of about 1 cm, parallel to the width.
2. Touch a drop of ink mixture at the centre of line drawn with the head of a pin. Allow the ink drop to dry on the filter paper for some time.
3. Fold the other end of paper on the glass rod upto about 1 cm length and then pin it as shown in the figure 5
4. Fill a measuring cylinder with water upto a height of 2 cm and on the mouth of cylinder place the glass rod with filter paper in such a way that the ink spot is above the water level.
5. Leave the apparatus undisturbed. The water level can be seen rising on the filter paper. Allow the water level to rise to the top of filter which takes about 20-30 minutes.
6. Take out the strip of paper. Mark the level to which the solvent (water) has travelled up to.
7. Then dry the strip having the coloured spots separated from the ink mixture. It

is known as chromatogram

8. Measure the distance from the top of the spots and that of the solvent from the initial line. Record them in the observation and results table. Repeat the experiment with any other solvent, if possible.

PRECAUTIONS

1. The glass rod should be horizontal
2. Water taken should be distilled water



5. . Separation of coloured substances by paper chromatography

OBSERVATION AND RESULT TABLE

| Nature of mixture | Distance travelled by (cm) | | | R _f values | |
|-------------------|----------------------------|-------------|--------------|-----------------------|----------|
| | Solvent (S) | Red ink (R) | Blue ink (B) | Red ink | Blue ink |
| Red and Blue ink | | | | | |

CALCULATIONS

Calculate the R_f value for each spot of a particular colour as follows (e g. for red ink)

R_f (red ink)

$$\frac{\text{Distance travelled by red spot}}{\text{Distance travelled by solvent (water)}}$$

CONCLUSION

The mixture is separated into , colours and R_f values for , colour is and for colour is

QUESTIONS

1. Compare your R_f values with each other and with specimens results from your coordinator. How do you explain some variation in results ?
2. Why do R_f values change when a different solvent is used ?

DETECTION OF NITROGEN, SULPHUR OR HALOGENS IN THE GIVEN ORGANIC COMPOUND

MAJOR SKILLS

1. Detection of nitrogen, sulphur or halogens in an organic compound.

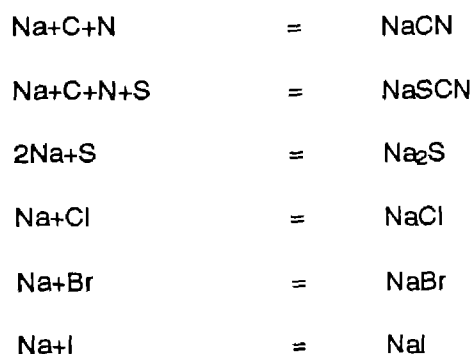
REQUIREMENTS

Ignition tubes, piece of sodium metal, pair of tongs, kerosene burner, China dish, distilled water, filter paper, wire gauge, glass rod, funnel, beaker (100 cm³), given organic compound, reagents as mentioned against the tests.

INTRODUCTION

In the qualitative analysis of inorganic salts, Experiment 12, you deal with ionic salts where different ionic species are present. But organic compounds are covalent where nitrogen, sulphur or halogens would be detected as constituent elements, if present. Carbon, Hydrogen are present in almost all the organic compounds, so there is no need to detect them. The covalently bonded elements are converted into anions by fusion with sodium metal. This process is called Lassaigne's Test

Thus



The sodium salts produced are water-soluble ionic compounds. These can, therefore, be detected by employing the usual tests for anions.

INSTRUCTIONS

Prepare sodium extract (Lassaigne extract) as follows :

1. Take a China dish and put about 10 cm³ distilled water in it.
2. Take a small piece of sodium with the help of a pair of tongs and dry it between two folds of a filter paper.
3. Drop the sodium piece into an ignition tube and heat it gently over the kerosene burner flame till the metal melts to a shining globule.
4. Introduce a small quantity of the given organic compound into the ignition tube and heat it until the lower end of the tube becomes red hot.

MAJOR SKILLS

Preparation of soap

REQUIREMENT

Boiling tube, Coconut oil, beaker (250 cm^3), sodium hydroxide, glass rod, water, kerosene burner, tripod stand, wire gauge, sodium chloride, thin muslim cloth

INTRODUCTION

Soaps are alkali metal salts (sodium and potassium) of higher fatty acids containing 12 to 18 carbon atoms. The sodium salts of acid (e.g. stearic, oleic acid, etc.) are soft semisolids and are used for washing purposes. The potassium salts are more like liquids and are called as soft soaps. These are used for bathing purposes. Soaps are prepared by the hydrolysis of oils and fats with caustic soda. Glycerine is obtained as bye-product in the process.

INSTRUCTIONS

1. Take about 10g of coconut oil (alternatively, mustard or ground nut oil can be used) in a boiling tube.
2. Dissolve 3g sodium hydroxide in 15 cm^3 water and mix it with the oil.
3. Take about 150 cm^3 of water in a 250 cm^3 beaker. Add the mixture from step 1 and 2 in the beaker. Stir the contents continuously with a glass rod.
4. Heat the contents of the beaker on wiregauge kept on tripod stand till greasy nature of mixture disappears. It takes about 30-40 minutes.
5. Prepare 100 cm^3 of a saturated sodium chloride solution in the water.
6. Prepare about $70\text{-}80\text{ cm}^3$ of saturated solution from step 5 to the hot soap mixture. Cool it for some time to precipitate the soap.
7. Filter it through a piece of thin muslin cloth and wash the precipitated soap with $15\text{-}20\text{ cm}^3$ of cold water.
8. Mould the soap into a cake in a small China dish.

PRECAUTIONS

1. As the reaction proceeds, mixture tends to acquire a paste form. So a continuous stirring is must for uniform mixing of contents.
2. The filtration of soap is done through a piece of thin cloth only. The use of filter paper should be avoided, else the soap will stick to it.

MAJOR SKILLS

1. Using the pipette and volumetric flask correctly
2. Preparation of a standard solution.

REQUIREMENTS

Oxalic acid, volumetric flask (100 cm^3), wash bottle, pipette, funnel, distilled water.

INTRODUCTION :

A solution of known concentration is termed as standard solution. The strength is expressed in moles per dm^3 . It is obtained by dissolving a known amount of a primary standard substance in a definite volume of a solvent.

A primary standard should

1. be in a highly pure state
2. be stable in air
3. be easily soluble
4. have a high molar mass
5. undergo stoichiometric and rapid reaction when used in volumetric analysis.

Oxalic acid and ferrous ammonium sulphate meet the above requirements, so these are primary standards.

INSTRUCTIONS :

1. Take a packet of oxalic acid. It contains 15g of oxalic acid.
2. Take about 50cm^3 of distilled water in volumetric flask.
3. Put a clean and dry funnel on the flask and carefully transfer all the oxalic acid to the flask, through the funnel.
4. If some oxalic acid is still remaining in the packet wash it carefully in the funnel with the help of wash bottle.
5. Shake the flask to dissolve all the oxalic acid. If it does not dissolve completely, add a little more of water.
6. Put more water through the funnel to the flask with the help of wash bottle, so that the level of water comes to about 2 cm below the mark on the neck of the flask.
7. Take clean pipette and fill it about half with distilled water and add a few drops of water into the flask till the meniscus just touches the graduation mark on the flask.
8. Stopper the flask and shake it to make the solution homogenous.

9. Calculate the molar concentration of the solution and label it

PRECAUTIONS :

1. Oxalic acid should be transferred completely. Any loss would affect the concentration.
2. Addition of water to the volumetric flask should be done carefully and in no circumstances should the level of water be allowed to over shoot the graduation mark (etched on the neck of the volumetric flask)

RESULT : The molarity of oxalic acid solution is

11b.

STANDARDISATION OF GIVEN SOLUTION OF KMnO_4 BY TITRATING IT AGAINST A STANDARD SOLUTION OF OXALIC ACID.

MAJOR SKILLS

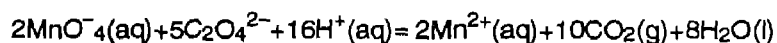
1. Performing a titration
2. Detection of end point
3. Calculating the molarity of the standardised solution

REQUIREMENTS

Burette, pipette, conical flasks, funnel, solution of oxalic acid, solution of KMnO_4 , laboratory stand with clamp and boss, Kerosene burner tripod, wire gauge, match box, tile, water, dil, H_2SO_4 .

INTRODUCTION

Standard solution of certain substances e.g. KMnO_4 cannot be prepared by dissolving a known amount of the substance in a known volume of the solvent. These are termed as secondary standards. To find the concentration of such solution, these are titrated with a suitable standard solution. This process is termed as standardisation. Standardisation of KMnO_4 is done by titrating against oxalic acid at about 60°C , according to the following equation :



The end point of the titration is indicated by permanent appearance of faint pink colour. The concentration of KMnO_4 is found by using the following equation based on the stoichiometry of the reaction.

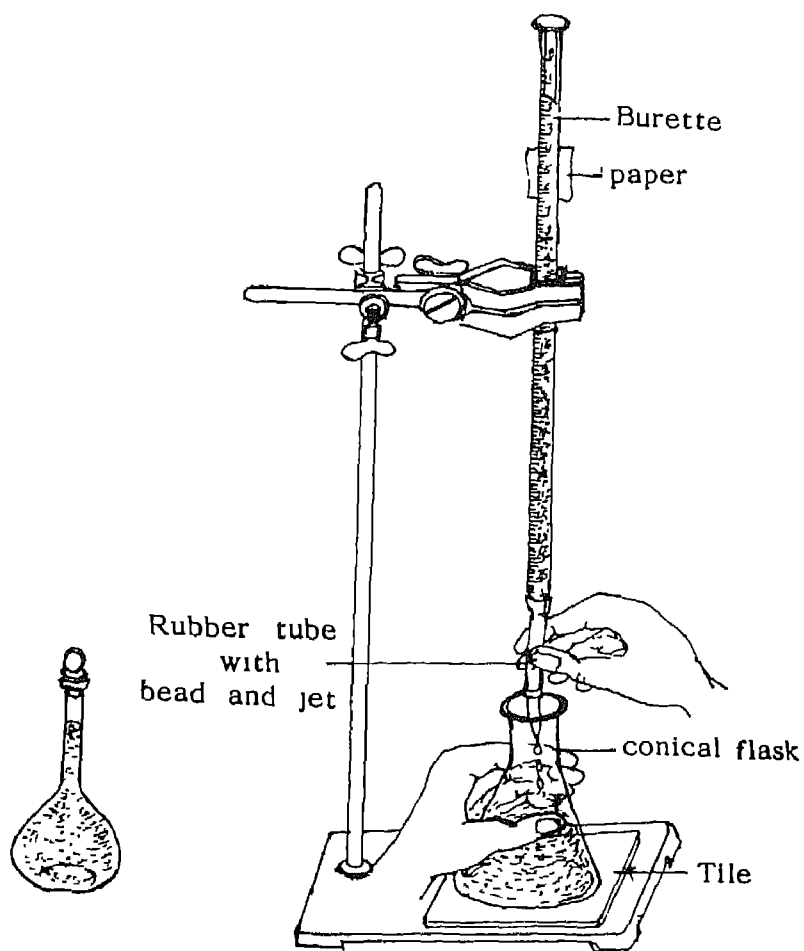
$$\frac{M_A V_A}{M_B V_B} = a/b$$

Where M_A is the molarity of oxalic acid and M_B is that of potassium permanganate and

- a are no. of moles of oxalic acid and
b are that of potassium permangnates.

INSTRUCTIONS

1. Take a clean burette and put about 5 cm^3 solution of KMnO_4 in it to rinse it
2. Fix the burette in the stand as shown in Fig. 6.
3. Discard this solution and fill the burette again with KMnO_4 .
4. Adjust the level of KMnO_4 in the burette by drawing out some of the solution. Make sure that no air gap remains in the plastic tube and nozzle (See



6. : Volumetric titration

instructions on how to use burette).

5. Read the level of liquid in the burette and record in the table under initial reading column.
6. Rinse the pipette with solution of oxalic acid and carefully transfer 10 cm^3 of oxalic acid solution in a clean 250 cm^3 conical flask.
7. Warm the solution of oxalic acid to about $60-70^\circ\text{C}$ over the burner (The flask should become just unbearable to touch).
8. Run solution of KMnO_4 from the burette in the flask slowly with continuous stirring until a permanent faint colour persists.
9. Read the level of the solution in the burette again and record in the table under the final reading column.
10. This reading may be used as a trial run, because you will probably overshoot the end point.
11. Repeat steps 2-8 atleast twice again, but this time add solution of KMnO_4 dropwise near the end point.
12. Record your observations in the table.

13. Make calculations using equation given above and report.

PRECAUTIONS

1. No air bubble/air gap should be present in the plastic tube and the glass nozzle
2. Burette readings must be carefully taken by reading upper meniscus.
3. Add KMnO_4 dropwise
4. Do not boil the solution of oxalic acid

OBSERVATION TABLE

| S. No. | Volume of oxalic acid (Cm ³) V ₁ | Burette Reading | | Volume of KMnO ₄ used, V ₂ (Final – Initial) (Cm ³) |
|--------|------------------------------------------------------------|-------------------------------|-----------------------------|---------------------------------------------------------------------------------------------|
| | | Initial (Cm ³) | Final (Cm ³) | |
| 1. | | | | |
| 2. | | | | |
| 3. | | | | |
| 4. | | | | |

CALCULATIONS

$$\text{(Oxalic Acid)} = \text{(KMnO}_4\text{)}$$

$$2 \times M_1 \times V_1 = 5 M_2 V_2$$

$$2 \times \frac{1}{10} \times V_1 = 5 M_2 V_2$$

V₁ and V₂ are known from the above table, M₂ can be calculated.

RESULT : Molarity of KMnO₄ solution is

QUESTION

At the end point, why does a permanent appearance of faint pink colour exist ?

**ELEMENTARY QUALITATIVE ANALYSIS OF A SALT INVOLVING
DETECTION OF ONE CATION AND ONE ANION****MAJOR SKILLS**

1. Observing physical properties of salts
2. performing wet tests
3. confirming the anion & cation present

REQUIREMENTS

Kipp's apparatus, burner, test tubes, test tube stand, test tube brush, test tube holder, spatula, delivery tubes, Nichrome wire, watch glass, boiling tube, beaker (100 cm³) tripod, wire gauge, funnel, filter paper, chemicals and reagents for qualitative analysis

INTRODUCTION

The inorganic qualitative analysis involves the identification of ions present in a sample. The salt consists of anion and cation. The basis of identification is the observed chemical behaviour of the components. The salt analysis may be systematically carried out in the following three steps:

- (i) Preliminary examination
- (ii) Detection of anions and
- (iii) Detection of cations

Preliminary examination :

First we proceed to examine the physical properties of the salt. This followed by the action of heat on the salts and also certain tests, such as flame test etc.

Detection of Anions :

A preliminary hint about some anions may be obtained by the formation of volatile or gaseous products when the salt is treated with

- (i) dil. H₂SO₄ (for CO₃²⁻ and NO₃⁻)
- (ii) conc. H₂SO₄ (for Cl⁻, Br⁻, I⁻ and NO₃⁻)

A confirmation is made by other specific tests. Other anions (SO₄²⁻ and PO₄³⁻) are identified by different reagents.

For performing some identification tests for anions, it is essential to prepare the salt solution. Depending upon the solubility of the given salt, we may prepare its aqueous solution or water extract or sodium carbonate extract for carrying out the tests.

Water Extract (WE)

Boil 1-2 g of the salt with 10-20 cm³ distilled water for 2-3 min.

Sodium Carbonate Extract (SE)

If the salt under test is insoluble in water, its sodium carbonate extract is prepared. On boiling the salt with sodium carbonate solution, double decomposition takes place resulting in the formation of soluble sodium salt of the anion. Mix 1-2g of the salt with 3-5g of Na₂CO₃ in a boiling tube. Add about 20 cm³ of distilled water and boil for about 10 min. Filter. The filtrate is the sodium carbonate extract (SE)

Detection of Cations

All cations may be divided into six groups depending upon the difference in solubility of their chlorides, sulphides, hydroxides, carbonates under different conditions of acidity and alkalinity. The reagent used to test the presence of a group is known as a group reagent.

The first step before proceeding to systematic wet analysis of cations is to dissolve the salt in a suitable solvent. The clear solution is known as original solution (OS)

INSTRUCTIONS

1. Note the colour, odour and state of the given salt as given in the table 1.

Table 1 : Physical properties of salts.

| PHYSICAL PROPERTIES | INFERENCE |
|--------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| White Colour (Colourless) | Pb ²⁺ , Al ³⁺ , Zn ²⁺ , Ca ²⁺ , Ba ²⁺ , Sr ²⁺ , Mg ²⁺ or NH ₄ ⁺ salt may be present |
| Bluish or bluish green colour | Cu ²⁺ or Ni ²⁺ salt |
| Light green colour | Fe ²⁺ or Ni ²⁺ salt |
| Yellowish brown colour | Fe ³⁺ salt |
| Smell of ammonia gas | NH ₄ ⁺ salt |
| Smell of hydrogen sulphide gas | S ²⁻ |
| Heavy | Pb ²⁺ , Ba ²⁺ salt |
| Light | Al ³⁺ , Mg ²⁺ , Ca ²⁺ salt |

2. Take a small quantity of the salt in a dry test tube with the help of a spatula. Heat the test tube over the burner and observe the changes. Note the colour, odour of the gases if evolved as given in table 2.
3. Prepare a paste of small amount of the salt with one or two drops of conc. HCl on a watch glass. Take the paste on the loop of Nichrome wire, put it in the burner's flame and observe the colour as given in table 3.

Table 2 : Action of Heat

| Observation | Inference | Chemistry Involved |
|------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|
| Salt appears as if melting | Presence of some hydrated salt | |
| Ammonia gas evolves | NH_4^+ | $(\text{NH}_4)_2\text{CO}_3 = \text{NH}_3 + \text{CO}_2 + \text{H}_2\text{O}$ |
| Carbondioxide evolves | CO_3^{2-} present | $\text{CaCO}_3 = \text{CaO} + \text{CO}_2$ |
| Sulphurdioxide evolves | SO_4^{2-} may be present | $2\text{FeSO}_4 = \text{Fe}_2\text{O}_3 + \text{SO}_2 + \text{SO}_3$ |
| Nitrogen dioxide (brown gas) | NO_3^- | $2\text{Cu}(\text{NO}_3)_2 = 2\text{CuO} + 4\text{NO}_2 + \text{O}_2$ |
| Colour of the salt changes from 1. blue to white 2. Green to yellow 3. White (when cold) to yellow (when hot) | Cu^{2+} salt Ni^{2+} salt Zn^{2+} salt | Some salts change their colour by losing water of crystallisation or due to change in state |

Table 3 : Flame Test

| Flame colour by naked eye | Inference |
|---------------------------|------------------|
| Deep blue green | Cu^{2+} |
| Brick red | Ca^{2+} |
| Apple green | Ba^{2+} |
| Crimson (deep red) | Sr^{2+} |

4. Perform tests for the anions with dil. H_2SO_4 as follows in table 4.

Table 4 : Action of dil. H_2SO_4

(Tests for CO_3^{2-} and S^{2-} radicals)

| Experiment | Observation | Inference | Chemistry Involved |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Take a small quantity of the salt in a test tube and add a few cm^3 of dil. H_2SO_4 . In case there is no change, heat the test tube | 1. Evolution of colourless gas with brisk effervescence. 2. Colourless gas with rotten eggs' smell of H_2S . | CO_3^{2-} S_2^{2-} | $\text{CaCO}_3 + \text{H}_2\text{SO}_4 = \text{CaSO}_4 + \text{CO}_2 + \text{H}_2\text{O}$ $\text{S}^{2-} + \text{H}_2\text{SO}_4 = \text{SO}_4^{2-} + \text{H}_2\text{S}$ |
| Confirmatory test for CO_3^{2-} Pass the issuing CO_2 gas through lime water or barium chloride solution. | Lime water turns black | CO_3^{2-} confirmed | $\text{Ca}(\text{OH})_2 + \text{CO}_2 = \text{CaCO}_3$ |
| Confirmatory tests for S^{2-} 1. Bring moist lead acetate paper near the evolving H_2S gas 2. Bring moist $\text{K}_2\text{Cr}_2\text{O}_7$ paper near the evolving gas 3. Add a few drops of sodium nitroprusside to the alkaline solution (made by adding NaOH of the sulphide) | Paper turns black The orange colour acquires light green color Violet coloration produced | S^{2-} confirmed S^{2-} confirmed S^{2-} confirmed | $(\text{CH}_3\text{COO})_2\text{Pb} + \text{H}_2\text{S} = \text{CH}_3\text{COOH} + \text{PbS}$ |

5. Take a small quantity of the salt in a test tube and, add about 2 cm³ of conc. H₂SO₄ warm the contents and make observations (do not boil) as given in table 5A

Table 5 A : Action of H₂SO₄
(Test for salt)

| Observation | Inference | Chemistry involved |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1. The gas evolved is colourless and pungent smelling | Cl ⁻ may be present | Cl ⁻ + H ₂ SO ₄ = HSO ₄ ⁻ + HCl |
| 2. A colourless gas is evolved a. brown coloured gas b. If fumes of the gas are intensified by the addition of copper turnings c. violet vapours with pungent odour | Br ⁻ or NO ₃ ⁻ may be present NO ₃ ⁻ I ⁻ | Br ⁻ + H ₂ SO ₄ = HSO ₄ ⁻ + HBr NO ₃ ⁻ + H ₂ SO ₄ = HSO ₄ ⁻ + HNO ₃ HNO ₃ + Cu = Cu(NO ₃) ₂ + NO ₂ I ⁻ + H ₂ SO ₄ = HSO ₄ ⁻ + HI |

6. Perform tests for anions Cl⁻, Br⁻, I⁻, NO₃⁻ with conc. H₂SO₄ as follows and carryout confirmatory tests for the radical inferred as given in table 5B.

Table 5 B : Action of H₂SO₄
(Confirmatory tests for Cl⁻, Br⁻, I⁻ and NO₃⁻)

| Experiment | Observation | Chemistry involved |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------|
| For Cl⁻ 1. When heated with conc. H ₂ SO ₄ HCl gas is given out. Bring a glass rod moistened at the tip with ammonia solution near the issuing gas. | Dense white fumes of NH ₄ Cl are produced | NH ₃ + HCl = NH ₄ Cl |
| 2. Acidify a small quantity of W.E. or S.E. in the dil. HNO ₃ and add AgNO ₃ solution | White curdy ppt of AgCl | AgNO ₃ + Cl ⁻ = AgCl |
| 3. Treat the white ppt with ammonia solution. | The ppt dissolves | AgCl + 2NH ₃ = [Ag(NH ₃) ₂]Cl |
| 4. Chromyl chloride test. Take a mixture of solid salt and potassium dichromate in a test tube and add a few cm ³ of conc. H ₂ SO ₄ to it. Heat the test tube carefully and let the issuing coloured gas bubble in NaOH solution. | Coloured gas yellow coloured solution | CrO ₂ Cl ₂ + 4NaOH = 2NaCl + 2H ₂ O + Na ₂ CrO ₄ |
| For Br⁻ 1. Manganese dioxide test. Mix MnO ₂ with a little of the salt and heat the mixture with conc. H ₂ SO ₄ | Brown fumes | 2NaBr + MnO ₂ + 2H ₂ SO ₄ = Na ₂ SO ₄ + Br ₂ + MnSO ₄ + 2H ₂ O |
| 2. Add AgNO ₃ solution to the acidified W.E. or S.E. | Light yellow ppt of AgBr | Br ⁻ + AgNO ₃ = AgBr + NO ₃ ⁻ |

| Experiment | Observation | Chemistry involved |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 3 Add NH_4OH to the AgBr ppt | ppt sparingly dissolves | |
| For I^- | | |
| 1. Manganese dioxide test | Violet vapours | $2\text{KI} + \text{MnO}_2 + 2\text{H}_2\text{SO}_4 = \text{MnSO}_4 + \text{I}_2 + \text{K}_2\text{SO}_4 + 2\text{H}_2\text{O}$ |
| 2. AgNO_3 test | Yellow ppt AgI | $\text{AgNO}_3 + \text{I}^- = \text{AgI} + \text{NO}_3^-$ |
| 3 Add NH_4OH to the AgI ppt | ppt insoluble | |
| For NO_3^- | | |
| 1. With copper turnings. Heat a mixture of the salt and conc H_2SO_4 and add a few copper turnings | Dense reddish brown fumes of NO_2 | $\text{NaNO}_3 + \text{H}_2\text{SO}_4 = \text{NaHSO}_4 + \text{HNO}_3$ $3\text{Cu} + 8\text{HNO}_3 = 3\text{Cu}(\text{NO}_3)_2 + 2\text{NO} + 4\text{H}_2\text{O}$ $2\text{NO} + \text{O}_2 = 2\text{NO}_2$ |
| 2. Ring test Take W.E. of the salt in a test tube and add to it freshly prepared solution of FeSO_4 . Now add conc H_2SO_4 along the sides of the test tube, containing the solutions held in a standing position | Formation of a black ring at the junction of two layers | $\text{FeSO}_4 + \text{NO} = \text{FeSO}_4 \cdot \text{NO}$ |

7. Carry out individual tests for SO_4^{2-} and PO_4^{3-} as given in the table 6.

Table 6 : Individual tests for SO_4^{2-} and PO_4^{3-} acid radicals

| Experiment | Observation | Chemistry involved |
|-------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------|-----------------------------------------------------|
| a. Tests for SO_4^{2-} Acidify the sodium carbonate extract with conc. HCl add BaCl_2 solution to it. | White ppt insoluble in conc. HCl | $\text{SO}_4^{2-} + \text{Ba}^{2+} = \text{BaSO}_4$ |
| b. Test for PO_4^{3-} Warm the salt with a few drops of conc. HNO_3 and then add excess of ammonium molybdate | yellow ppt | |

8. Carry out wet tests for cations. Follow the systematic procedure for identification of cations as given below :

- Test for zero group (NH_4^+) first
- Prepare original solution and add respective group reagents (Table 7)

Tests for NH_4^+ radical

- Take a small quantity of the salt in a test tube and add a few cm^3 of NaOH solution. Test the vapour for ammonia gas by smell and by bringing a drop of conc. HCl at the tip of a glass rod to the mouth of the test tube. (Dark white fumes of NH_4Cl)
- Pass the evolved ammonia gas through a tube containing a few cm^3 of Nessler's reagent. Formation of a reddish brown ppt. confirms NH_4^+ .

Table 7 : Schematic separation of cations into groups

| Add dil HCl to the original solution (O.S.) | | | | | |
|---------------------------------------------|--------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------|----------------------------------------------------------------|-----------------------------------------|
| Precipitate Pb^{2+} | Clear solution. Pass H_2S gas into the acid solution Dilute the clear solution and again pass H_2S | | | | |
| | Precipitate Coloured Group II Pb^{2+} , Cu^{2+} , Cd^{2+} | Clear solution. Boil to expel H_2S (o take fresh sample of O.S.) Add conc HNO_3 and boil. Add solid NH_4Cl and a slight excess of NH_4OH | | | |
| | | Precipitate Group III Fe^{3+} , Al^{3+} | Clear solution. Pass H_2S in basic medium | | |
| | | | Precipitate Group IV Zn^{2+} , Ni^{2+} , Mn^{2+} | Clear solution | |
| | | | | Precipitate Group V Ba^{2+} , Sr^{2+} , Ca^{2+} | Clear solution Group VI Mg^{2+} |

Preparation of original solution (O.S.)

Take a small amount of the salt in a boiling tube :

- Add a few cm^3 of distilled water and shake it. If it is not soluble, warm it.
- If salt is insoluble, add dil HCl (cold/hot) and obtain a clear solution.

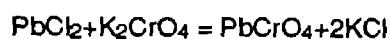
Label the clear solution O.S.

- Perform confirmatory tests for cation from the respective group, precipitate as given below :

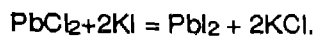
Group I $PbCl_2$

Confirmatory tests

- The precipitate is soluble in hot water and reappears on cooling.
- Add K_2CrO_4 solution to a warm solution of $PbCl_2$ yellow precipitate of $PbCrO_4$.



- Add KI solution to a warm solution of $PbCl_2$. Yellow ppt. of PbI_2 .



Group II Black : PbS , CuS

Yellow : CdS

Black ppt

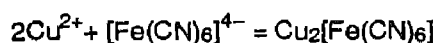
Heat with 50% HNO_3 (1:1) in a test tube.

- Black precipitate dissolves giving bluish green solution (Cu^{2+}).

Confirmatory tests for Cu^{2+}

Acidify blue solution with acetic acid and add $K_4[Fe(CN)_6]$ solution. A chocolate

brown precipitate appears.



2. Black ppt dissolves giving colourless solution Pb^{2+}

Confirmatory tests for Pb^{2+}

To the lead solutions, add a little alcohol and dil. H_2SO_4 in excess. A white ppt is formed. Dissolve the ppt. in ammonium acetate solution. Divide into two parts.

To part I, add KI solution - Yellow ppt. (PbI_2)

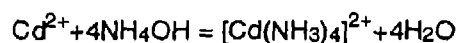
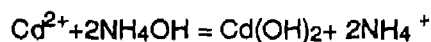
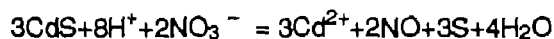
To part II, add K_2CrO_4 solution - Yellow ppt (PbCrO_4)

Yellow ppt :

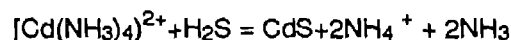
Dissolve the ppt by warming with dil. HNO_3 .

Confirmatory test for Cd^{2+} :

- (i) Add NH_4OH dropwise. A white ppt. soluble in excess



- (ii) Through above solution pass H_2S in excess. A yellow ppt conforms Cd^{2+}



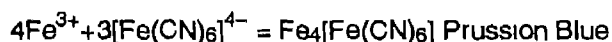
Group III $\text{Fe}(\text{OH})_3$: Brown ppt

$\text{Al}(\text{OH})_3$: White gelatinous

Confirmatory test (Fe^{3+})

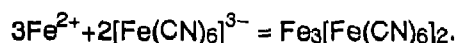
Dissolve the ppt. in dil. HCl and divide into two parts.

- (i) To part I, add $\text{K}_4[\text{Fe}(\text{CN})_6]$ solution. An intense blue colour or ppt. confirms Iron.



Distinction between Ferrous (Fe^{2+}) and Ferric (Fe^{3+})

- (i) Add Pot. ferricyanide to the O.S. Deep blue colouration or ppt. indicates Fe^{2+} while brown colouration indicates Fe^{3+} .



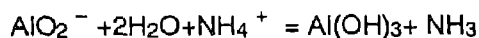
- ii) Add Pot. ferrocyanide solution to the O.S. Deep blue coloration or ppt. indicates Fe^{3+} .

White Gelatinous ppt.

Confirmatory tests (Al^{3+})

Dissolve the ppt. by boiling with NaOH solution.

- (i) Add NH_4Cl in excess. Boil. A white gelatinous ppt.



Group IV

Black ppt : NiS

White ppt. : ZnS

Buff coloured ppt. : MnS

Black ppt (NiS)

Solution for the fourth group is of green colour

Confirmatory tests (Ni^{2+})

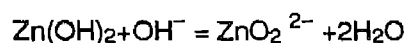
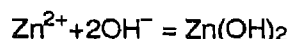
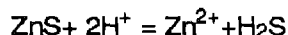
Dissolve the black ppt in aquaregia ($\text{HCl} + \text{HNO}_3$). Add 1-2 drops of dimethylglyoxime solution and NH_4OH to alkaline. A scarlet red ppt. is obtained.

White ppt (ZnS)

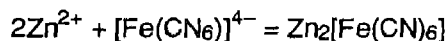
Confirmatory test (Zn^{2+})

Dissolve ppt. in dil. HCl . Boil off H_2S and divide into two parts :

- i) To part I, add NaOH dropwise. A white ppt which dissolves in excess.



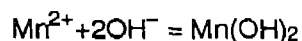
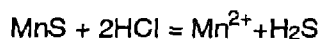
- ii) To part II, add $\text{K}_4[\text{Fe}(\text{CN})_6]$ solution. A white ppt.



Buff Coloured ppt (MnS)

Confirmatory tests

- i) Dissolve ppt. in dil. HCl , boil off H_2S . Add NaOH in excess. A white ppt. turning brown on standing.



$\text{Mn}(\text{OH})_2$ turns brown on standing due to its oxidation into black MnO_2 and finally becomes hydrated i.e. $\text{MnO}_2 \cdot \text{H}_2\text{O}$

Group V BaCO_3 , SrCO_3 , CaCO_3

Dissolve the ppt. in warm acetic acid. Boil off CO_2 , Divide into 3 parts :

Confirmatory test for Ba^{2+}

To one part of the above solution, add K_2CrO_4 solution. A yellow ppt. of BaCrO_4

Filter and make a paste of this ppt. with conc. HCl and apply flame test. A green flame.

Confirmatory test for Sr^{2+}

If Ba^{2+} is absent then to part II, add $(\text{NH}_4)_2\text{SO}_4$, warm. A white ppt.

Filter white ppt, make a paste with conc. HCl . Apply flame test. A crimson red flame.

Confirmatory test for Ca^{2+}

Ba^{2+} and Sr^{2+} are absent, then to part III add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. A white ppt.

Filter and make a paste of this ppt. with conc. HCl . Apply flame test. A brick red flame.

Group VI (Mg^{2+})

To the Mg^{2+} solution, add NH_4Cl and NH_4OH to alkaline, then add excess of ammonium phosphate solution. Scratch the sides of the test tube with a glass rod. A white ppt.

PRECAUTIONS

1. Proceed with small quantity of the salt only.
2. Never boil concentrated acid solutions. Warm them.
3. Never throw hot concentrated acids into the sink.

OBSERVATIONS AND RESULTS TABLE

[illegible]

| EXPERIMENTS | OBSERVATIONS | INFERENCE |
|-------------------------------------|--------------|-----------|
| Confirmatory test for basic radical | | |

RESULT

The given salt contains :

Cation Anion

QUESTIONS

1. What do you mean by wet tests ?
2. Why do you prepare sodium carbonate extract ?
3. What is a group reagent ?
4. Why copper is precipitated in the second group whereas Zinc is precipitated in the fourth group.
5. Why is it essential to boil off H_2S completely before proceeding to group III ?
6. Why do we add ammonium chloride before adding ammonia solution to precipitate group III cations ?

